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Revision No. 0

QUALITY ASSURANCE PROJECT PLAN
FOR
SPRINGFIELD IRON
REMOVAL ACTION SITE
SPRINGFIELD, SANGAMON COUNTY, ILLINOIS
NPL STATUS: NON-NPL

Prepared for

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

by

WESTON SOLUTIONS, INC.

October 3, 2013(Revision 0)

EPA Contract No. EP-S5-06-04
TDD Number: S05-0001-1307-013

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QAPP ELEMENTS

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QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name: Springfield Iron Removal Site
Site Location: 1900 Factory Street, Springfield, Sangamon County, Illinois

Quality Assurance Project Plan (QAPP) for the Springfield Iron Removal Site
Document Title

United States Environmental Protection Agency Region V
Lead Organization

Jeff Bryniarski, Weston Solutions, Inc. (WESTON®) Superfund Technical Assessment and Response Team (START)
Preparer's Name and Organizational Affiliation

20 North Wacker Drive, Suite 2035, Chicago, Illinois, 60606, 312-424-3300, jeff.bryniarski@westonsolutions.com
Preparer's Address, Telephone Number, and E-mail Address

October 3, 2013
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager:_____
Signature

Tonya Balla WESTON START, October 3, 2013
Printed Name/Organization/Date Printed

Investigative Organization's Project QA Officer:_____
Signature

Lisa Graczyk WESTON START, October 3, 2013
Printed Name/Organization/Date

Lead Organization's Project Manager:_____
Signature

Jaime Brown, EPA, October 3, 2013
Printed Name/Organization/Date

Approval Signatures:_____
Signature

_____
Printed Name/Title/Date

_____
Approval Authority

Other Approval Signatures:_____
Signature

_____
Printed Name/Title/Date

Document Control Number: 2228-2E-BJNG

QAPP Worksheet #2
QAPP Identifying Information

Site Name/Project Name: Springfield Iron Removal Site
Site Location: 1900 Factory Street, Springfield, Sangamon County, Illinois
Site Number/Code: C5D8
Operable Unit: Not Applicable (NA)
Contractor Name: Weston Solutions, Inc.
Contractor Number: EP-S5-06-04
Contract Title: Superfund Technical Assessment and Response Team
Work Assignment Number: S05-0001-1307-013

1. Identify guidance used to prepare QAPP:
   Uniform Federal Policy for Quality Assurance Project Plans

2. Identify regulatory program:
   EPA Region V, Emergency Response Branch

3. Identify approval entity:
   EPA Region V

4. Indicate whether the QAPP is a generic or a project-specific QAPP. (circle one)

5. List dates of scoping sessions that were held:
   The scoping meeting was conducted concurrent with the site walkthrough on September 19, 2013. Jaime Brown (EPA), Jeff Bryniarski (WESTON START), and Toby Viehweg (Environmental Restoration) were present during scoping meeting.

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	**Approval Date**
Not Applicable	

7. List organizational partners (stakeholders) and connection with lead organization:
   Illinois Environmental Protection Agency (IEPA) – Bruce Everetts

8. List data users:
   EPA Region V, On-Scene Coordinator (OSC)

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

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COC – Chain-of-Custody
PQO – Project Quality Objectives
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
SOP – Standard Operating Procedure

QAPP Worksheet #3
Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	DCN
Jaime Brown	OSC	EPA	312-886-2256	312-353-9176	brown.jaime@epa.gov	2228-2E-BJNG
Tonya Balla	Project Manager	WESTON	847-918-4094	847-918-4055	t.balla@westonsolutions.com	2228-2E-BJNG
Lisa Graczyk	Project QA Officer	WESTON	312-424-3339	312 424-3301	lgraczyk@css-dynamac.com	2228-2E-BJNG
Jeff Bryniarski	Site Leader	WESTON	312-424-3307	312-424-3330	jeff.bryniarski@westonsolutions.com	2228-2E-BJNG
Toby Viehweg	ERRS Response Manager and Sample Management	ER	312-446-6325	NA	t.viehweg@erllc.com	2228-2E-BJNG

Notes:
DCN – Document Control Number
ER – Environmental Restoration
ERRS – Emergency and Rapid Response Services
NA – Not Available
OSC – On-Scene Coordinator
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
WESTON – Weston Solutions, Inc.

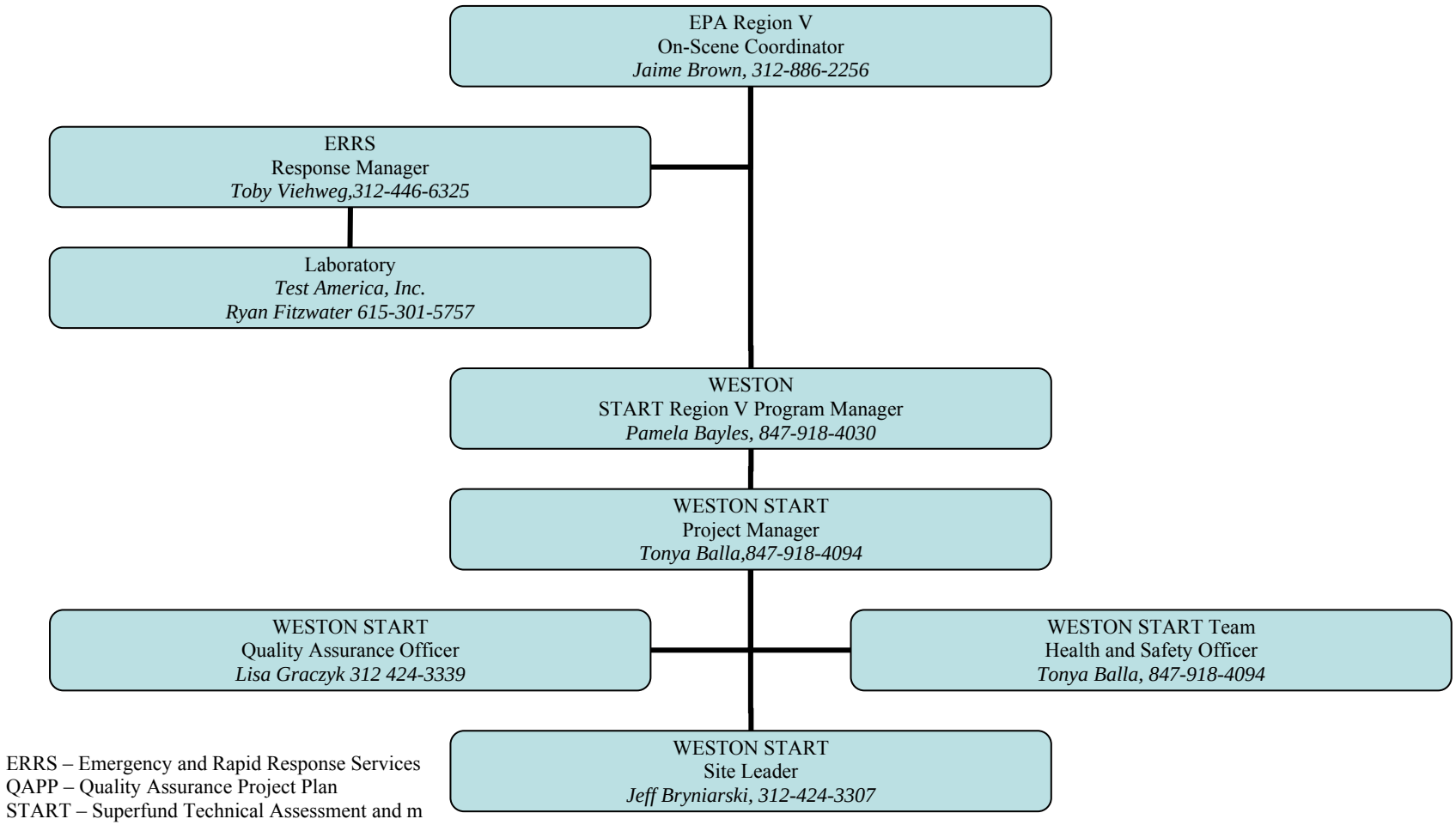
QAPP Worksheet #4
Project Personnel Sign-Off Sheet

Organization: Weston Solutions, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jaime Brown	EPA OSC	312-886-2256		
Jeff Bryniarski	QAPP Preparer	312-424-3307		
Tonya Balla	PM and Project QA Officer	847-918-4094		
Toby Viehweg	ERRS RM and Sample Management Coordinator	312-446-6325		

Notes:
ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
RM – Response Manager

QAPP Worksheet #5
Project Organizational Chart



ERRS – Emergency and Rapid Response Services
QAPP – Quality Assurance Project Plan
START – Superfund Technical Assessment and m
WESTON – Weston Solutions, Inc.

QAPP Worksheet #6
Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Project scope changes	EPA OSC	Jaime Brown	312-886-2256	The OSC will inform the WESTON PM of any project scope changes. The WESTON PM will in turn inform the START Program Manager of the changes.
Management of required START project tasks	PM	Tonya Balla	847-918-4094	The WESTON PM will inform the appropriate WESTON project staff (field and non-field) of tasks to complete and the required completion date. The WESTON project staff will communicate with the PM of task progress and resources/information required to complete tasks.
Delays or changes to field work	Site Leader	Jeff Bryniarski	312-424-3307	The site leader will inform the PM of any delays or changes to field work by telephone. The PM or Site Leader will inform the OSC by telephone.
Daily field updates	Site Leader	Jeff Bryniarski	312-424-3307	The site leader will inform the PM of daily field progress by telephone. The site PM (or site leader as the PM's designee) will inform the OSC of field work progress by telephone, email, or direct communication in the field.
Reporting of Laboratory Data Quality Issues	ERRS SMC/ PM	Toby Viehweg/ Tonya Balla	312-446-6325/ 847-918-4094	The ERRS SMC or the PM will inform the OSC of any issues related to data quality upon receipt of samples or during analyses.
Recommendations to stop work and initiation of corrective actions	QA Officer-PM/ EPA OSC	Tonya Balla/ Jaime Brown	847-918-4094/ 312-886-2256	The QA Officer, PM, and OSC all have the authority to stop work and initiate corrective actions should there be a reason to do so. Whoever stops the work or initiates corrective actions will inform the Site Leader and PM immediately. The PM will ensure that the QA Officer and OSC are informed of the stop work and corrective actions.
Distribution of analytical data	ERRS SMC	Toby Viehweg	312-446-6325	The ERRS SMC will receive all deliverables from the laboratory and distribute them to the OSC, who will in-turn distribute the data to WESTON and any other interested parties.
Approval of QAPP Amendments	EPA OSC	Jaime Brown	312-886-2256	Approval of all QAPP amendments will be by the OSC prior to the changes being implemented.

Notes:
ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator

PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
SMC – Sample Management Coordinator
START – Superfund Technical Assessment and Response Team
WESTON – Weston Solutions, Inc.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Jaime Brown	EPA OSC	EPA Region V	The OSC has overall project authority and directs the project manager regarding the tasks required to meet project objectives. The OSC is also responsible for reviewing and approving the project-specific QAPP (and any amendments) prior to its implementation.	Federal OSC
Pamela Bayles	Program Manager	WESTON START Team	The START Program Manager is responsible for ensuring the quality of work performed under the Region V START III contract. The START Program Manager interfaces directly with the EPA Contracting Officer and Project Officer, and has overall responsibility and direction for task assignments.	M.E.M. (Masters in Environmental Management), Air and Water Resources; B.S., Biology; over 18 years experience
Tonya Balla	PM	WESTON START Team	The PM is responsible for managing all aspects of the project, WESTON project personnel, and subcontractors. The PM interfaces directly with the OSC regarding all project tasks.	B.S. Environmental Engineering; over 20 years experience
Lisa Graczyk	QA Officer	WESTON START Team	The QA Officer reviews the project QAPP and has overall responsibility for project QA. The QA Officer will also perform a compliance check of all data received from the laboratory.	B.S. Chemistry; over 20 years experience
Tonya Balla	H&S Officer	WESTON START Team	The H&S officer approves the HASP and provides guidance to field personnel on H&S issues.	B.S. Environmental Engineering; over 20 years experience
Toby Viehweg	SMC	ERRS	The SMC is responsible for the procurement of the laboratory and is the main interface with the laboratory regarding project deliverables and QA/QC aspects of the analyses. The OSC and WESTON interface with the laboratory through the SMC. The SMC also coordinates sample delivery, and ensures that all analyses are performed and results are delivered on time.	TBD
Jeff Bryniarski	Site Leader	WESTON START Team	The site leader manages the field team and all work performed in the field. The site leader interfaces directly with the project manager regarding field tasks and any issues that arise while in the field.	B.S. Environmental Engineering; over 5 years experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Jeff Bryniarski	QAPP Preparer	WESTON START Team	The QAPP preparer is responsible for preparing the site-specific QAPP and is in close communication with the PM, SMC, and QA officer regarding all aspects of project-specific requirements.	B.S. Environmental Engineering; over 5 years experience

Notes:
ERRS – Emergency and Rapid Response Services
H&S – Health and Safety
HASP – Health and Safety Plan
OSC – On-Scene Coordinator
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
SMC – Sample Management Coordinator
START – Superfund Technical Assessment and Response Team
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #8
Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates[1]
Field Sampling Activities	40-Hour OSHA HAZWOPER Training and Recurrently Annual 8-hour refreshers	WESTON	Various	Site Leader and Field Team Members	Jeff Bryniarski/TBD	WESTON's web-based EHS Track

Notes:
EHS – Environmental Health and Safety
HAZWOPER – Hazardous Waste Operations and Emergency Response
OSHA – Occupational Safety and Health Administration
QAPP – Quality Assurance Project Plan
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #9
Project Scoping Session Participants Sheet

Project Name: Springfield Iron Removal Site **Projected Date(s) of Sampling:** September 23, 2013 through October 31, 2013 **Project Manager:** Tonya Balla, WESTON	**Site Name:** Springfield Iron Removal Site **Site Location**: 1900 Factory Street, Springfield, Sangamon County, Illinois

Date of Session: September 23, 2013
Scoping Session Purpose: Scope of Work for Removal Action

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Jaime Brown	EPA OSC	EPA Region V	312-886-2256	brown.jaime@epa.gov	EPA OSC
Tonya Balla	PM	WESTON	847-918-4094	t.balla@westonsolutions.com	PM
Toby Viehweg	RM	ER	312-446-6325	t.viehweg@erllc.com	RM

Notes:
ER – Environmental Restoration
OSC – On-Scene Coordinator
PM – Project Manager
QAPP – Quality Assurance Project Plan
RM – Response Manager
WESTON – Weston Solutions, Inc.

Comments/Decisions: During the removal action, coal tar solids and contaminated soil is to be excavated, and shipped off site for disposal. The remedial cleanup goal is to be 800 mg/kg total lead, 1,300 mg/kg benzene, 15,000 mg/kg vanadium, and varies for PAHs (see Worksheet #15) as the site is slated for industrial use. The cleanup goal is based on EPA Removal Management Levels and the IEPA TACO remediation objective. Following excavation, confirmation samples will be collected to confirm that cleanup goals were achieved.

Action Items: Create a QAPP for the confirmation sampling.

Consensus Decisions: Compare to RMLs.

QAPP Worksheet #10
Problem Definition

The problem to be addressed by the project: The Springfield Iron Site is located at 1900 Factory Street in Springfield, Sangamon County, Illinois. The Site is located in a mixed commercial and residential area. The Springfield Iron Company originally occupied approximately 50 acres on the north side of Springfield, east of Factory Street and south of Griffiths Avenue. The Springfield Iron Company was north of what is now Ridgely Avenue and mostly west of what is now 14th Street. A portion of the Springfield Iron Company extended east to the present location of the Chicago and Illinois Railroad.

The Springfield Iron Company operated from 1872 until 1905, when the plant structures were demolished and the Site was leveled. The plant primarily manufactured iron and steel for the railroad industry. A puddle mill was established at the Site in June 1872, and the first iron rail was completed in September 1872. Later operations expanded to include production of both iron and steel rails, bar iron, fish plates, and track bolts. The iron and steel manufacturing processes used four "gas houses" that produced gas using coal, which was then either used for production or sold to the city to fuel street lights. On March 11, 2010, the Illinois Environmental Protection Agency (IEPA) Office of Site Evaluation conducted a pre-Comprehensive Environmental Response, Compensation, and Liability Information System (CERCLIS) screening assessment at the Site. The assessment was prompted by the observation of coal tar seeping onto a public sidewalk and city right-of-way in the northwest region of the Site. The sidewalk links a nearby high school, baseball/softball fields, a community park, and several residential areas. The screening assessment focused on four coal gas houses and on a potential coal-tar disposal area.

In October 2010, as part of the pre-CERCLIS screening assessment, IEPA mobilized a Geoprobe to drill 20 borings at the Site in the four gas house areas and the potential coal-tar disposal area. The boring depths ranged from 4 to 12 feet below ground surface (bgs). According to the pre-CERCLIS screening assessment report, the following compounds exceeded EPA Removal Action Levels (RAL): 2,4-Dimethylphenol, 3- and 4-Methylphenol, Benzo(a)anthracene, Benzo(a)pyrene, and Benzene. On February 9, 2012, the EPA received a request from the IEPA to conduct a time-critical removal assessment at the Site to document the need for EPA involvement to address imminent and substantial threats to the public health or welfare of the United States or the environment posed by Site-related conditions.

On August 28-29, 2012, the EPA and Weston START conducted a site assessment at the Site. During the site assessment, the on-site coal-tar seep areas were not fenced or secured. One coal-tar seep was on a public sidewalk, and seeps have been reported on public streets. Nearby human populations and animals could easily contact coal tar seeping out of the ground through public walkways or by trespassing. The following hazardous substances in soils and surficial coal tar at levels that exceed their respective removal management levels (RML): 2-methylnaphthalene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, dibenz(a,h)anthracene, dibenzofuran, indeno(l,2,3-cd)pyrene, and naphthalene. This time-critical removal action is being conducted to mitigate endangerment posed to human health, human welfare, and the environment by Site conditions.

The environmental questions being asked: There are two environmental questions to be asked during the soil excavation at the Springfield Iron Site. Was the cleanup objective of 800 mg/kg total lead, 1,300 mg/kg benzene, 15,000 mg/kg vanadium, and varies for PAHs (see Worksheet #15) in soil achieved? In addition, was the coal tar solids removal process sufficient to reduce hazardous substances in the soil?

The possible classes of contaminants and the affected matrices: Total lead, benzene, vanadium, and PAHs, in soil.

The rationale for inclusion of chemical and non-chemical analyses: Coal tar was found at the Springfield Iron site. The contaminants of concern are lead, vanadium, benzene, and PAHs in soil based on the former site investigations conducted in August 2012.

Project decision conditions ("If..., then..." statements):

If confirmation soil sample results exceed 800 mg/kg total lead, 1,300 mg/kg benzene, 15,000 mg/kg vanadium, or varies for PAHs (see Worksheet #15), then additional soil excavation will need to be conducted.

Notes:
CERCLIS – Comprehensive Environmental Response, Compensation, and Liability Information System
ERRS – Emergency and Rapid Response Services
IEPA – Illinois Environmental Protection Agency
mg/kg – Milligram per kilogrtam
QAPP – Quality Assurance Project Plan
RML – removal management levels
START – Superfund Technical Assessment and Response Team
WESTON – Weston Solutions, Inc.

QAPP Worksheet #11
Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? EPA Region V.

What will the data be used for? The data will be used to confirm that soil cleanup objectives were achieved.

What type(s) of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques): Soil samples collected during the removal action will be submitted to an offsite laboratory for the following analyses: total lead, vanadium, benzene, and PAHs. Analytical results will be compared to (1) the cleanup value for lead in industrial areas of 800 mg/kg lead (this is both the EPA Removal Management Level value and the IEPA TACO Tier I value for industrial soil) and (2) the cleanup value for vanadium in industrial areas of 15,000 mg/kg vanadium (EPA RML for industrial soil) and (3) the cleanup value for benzene in industrial areas of 1,300 mg/kg benzene (EPA RML for industrial soil). The ERRS contractor will procure the laboratory.

How "good" do the data need to be in order to support the environmental decision? The reporting limits need to be sufficient to compare results to 800 mg/kg total lead, 15,000 mg/kg vanadium, 1,300 mg/kg benzene, and varies for PAHs (see Worksheet #15). EPA-approved methods will be utilized (such as SW846 methods) and must be followed by the laboratory.

How much data are needed? (number of samples for each analytical group, matrix, and concentration): It is estimated that approximately 15 soil samples will be collected to confirm cleanup goals were reached during excavation. At a minimum, one sample will be collected for each 400 square foot area excavated at a surface depth of approximately 0 to 6 feet below ground surface (bgs). The cleanup confirmation samples will be analyzed for total lead, vanadium, and benzene.

Where, when, and how should the data be collected/generated? The soil confirmation sampling will occur after completion of the soil excavation areas which is expected to occur around September 23, 2013. Additional excavation soil confirmation sampling will occur in October, 2013.

Who will collect and generate the data? The ERRS and WESTON START contractors to EPA Region V.

How will the data be reported? The soil sample results will be reported by the ERRS-procured commercial laboratory in a summary report submitted to the ERRS Sample Management Coordinator by e-mail. The ERRS Sample Management Coordinator will distribute the summary report to the EPA OSC, who will in turn distribute the summary report to WESTON and any other interested parties. The WESTON QA Officer will perform a compliance check of all data received from the laboratory.

How will the data be archived? WESTON will maintain a copy of all site-related data and files for a period of 10 years in accordance with its policies. In addition, WESTON will give a copy of all data to the EPA OSC, who will archive the data in the EPA's records center.

Notes:

bgs – below ground surface
ERRS – Emergency and Rapid Response Services
Mg/kg – Milligrams per kilogram
OSC – On-Scene Coordinator
PAH – Polynuclear Aromatic Hydrocarbon
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
RML – Removal Management Level
START – Superfund Technical Assessment and Response Team
WESTON – Weston Solutions, Inc.

QAPP Worksheet #12
Measurement Performance Criteria Table

Matrix	Soil
Analytical Group[1]	total lead, vanadium, benzene, and PAHs
Concentration Level	High/Medium/Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
See Worksheet #17	LSOP-1, LSOP-2, LSOP-3	Overall Precision	RPD ≤ 50%	Field Duplicates	S&A
		Laboratory Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Overall Accuracy/Bias	80 to 120 %Recovery	Laboratory Control Samples	A
		Accuracy/Bias Contamination	Any detection of target analytes in the blank	Laboratory Blanks	A
		Representativeness	NA	Adherence to surface soil sampling procedures and quantity of samples to collect	S
		Sensitivity	Quantitation limit at MDLs for individual metals compounds	Method Detection Limit Study	A
		Completeness	90% of samples collected and analytical data received	Project manager assesses completeness of samples collected; laboratory project manager assess completeness of analytical requirements per the QAPP	S&A

Notes:
[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

% - Percent
≤ – Less than or equal to
MDL – Method Detection Limit
NA – Not Applicable
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
QC – Quality Control
RPD – Relative Percent Difference
SOP – Standard Operating Procedure

QAPP Worksheet #13
Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/Collection Dates)	How Data Will Be Used	Limitations on Data Use
Not Applicable				

Notes:
QAPP – Quality Assurance Project Plan

QAPP Worksheet #14
Summary of Project Tasks

Sampling Tasks:
1. Excavation confirmation sampling for lab analyses.

Analysis Tasks:
An ERRS-procured commercial laboratory will prepare and process soil samples for the following:

1. Metals totals- lead and vanadium (SW-846 Method 6010B/C)
2. Total benzene (SW-846 Method 8260B/C)
3. PAHs (Method 8270C/D)

Quality Control Tasks:
1. Collect field duplicate samples per QAPP.
2. Perform sample collection procedures per Worksheet #17
3. Laboratory to perform laboratory QC procedures. QC procedures include analyzing blanks, laboratory control sample, and matrix spike samples.
4. The WESTON QA Officer will perform a compliance check of all data received from the laboratory.

Secondary Data:
Not applicable.

Data Management Tasks:
Compare soil confirmation sample results to the industrial soil cleanup value (EPA RML).

Documentation and Records:
Confirmation sampling locations will be documented and all sample collection data will be recorded in field logbooks. COCs, air bills, and sample logs will be prepared and retained for each sample. A copy of all finalized documents and analytical data will be retained in a central file area.

Assessment/Audit Tasks:
Assessment of field activities will be carried out by the Project Manager through frequent contact with the site leader. Audits will be carried out as directed and approved by the EPA OSC.

Data Review Tasks:
The laboratory will review all analytical data for completeness and quality. The analytical data will then be submitted to the EPA's ERRS contractor for distribution to the EPA OSC. The EPA OSC will in turn distribute the analytical data to WESTON and any other parties. A case narrative describing any quality control issues with the analyses will be submitted with the final data report. In addition, the laboratory will qualify data in accordance with its quality policies. The WESTON QA Officer (or her designee) will also perform a compliance check of all data received from the laboratory.

Notes:
COC – Chain of Custody
ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
PAH – Polynuclear Aromatic Hydrocarbon
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
RML – Removal Management Level
SOP – Standard Operating Procedure
WESTON – Weston Solutions, Inc.

QAPP Worksheet #15
Reference Limits and Evaluation Table

Matrix	Soil
Analytical Group[1]	total lead, vanadium, benzene, and PAHs
Concentration Level	High/Medium/Low

Analyte	CAS Number	Project Action Limit	Project Quantitation Limit	Analytical Method[1]		Achievable Laboratory Limits[2]	
				MDLs	Method QLs	MDLs	QLs
Lead	7439-92-1	800 mg/kg	10 mg/kg	---	---	0.70 mg/kg	1 mg/kg
Vanadium	7440-62-2	15,000 mg/kg	10 mg/kg	---	---	3.1 mg/kg	10 mg/kg
Benzene	71-43-2	1,300 mg/kg	10 mg/kg	---	---	0.00067 mg/kg	0.002 mg/kg
Acenaphthene	83-32-9	99,000 mg/kg	10 mg/kg	---	---	0.01 mg/kg	0.067 mg/kg
Acenaphthylene	208-96-8	---	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Anthracene	120-12-7	500,000 mg/kg	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Benzo[a]anthracene	56-55-3	210 mg/kg	10 mg/kg	---	---	0.015 mg/kg	0.067 mg/kg
Benzo[a]pyrene	50-32-8	21 mg/kg	10 mg/kg	---	---	0.012 mg/kg	0.067 mg/kg
Benzo[b]fluoranthene	205-99-2	210 mg/kg	10 mg/kg	---	---	0.012 mg/kg	0.067 mg/kg
Benzo[g,h,i]perylene	191-24-2	---	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Benzo[k]fluoranthene	207-08-9	210 mg/kg	10 mg/kg	---	---	0.014 mg/kg	0.067mg/kg
Chrysene	218-01-9	21,000 mg/kg	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Dibenz(a,h)anthracene	226-36-8	21 mg/kg	10 mg/kg	---	---	0.007 mg/kg	0.067 mg/kg
Fluoranthene	206-44-0	66,000 mg/kg	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Fluorene	86-73-7	66,000 mg/kg	10 mg/kg	---	---	0.012 mg/kg	0.067 mg/kg
Indeno[1,2,3-cd]pyrene	193-39-5	210 mg/kg	10 mg/kg	---	---	0.010 mg/kg	0.067 mg/kg
Naphthalene	91-20-3	1,800 mg/kg	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Phenanthrene	85-01-8	---	10 mg/kg	---	---	0.009 mg/kg	0.067 mg/kg
Pyrene	129-00-0	50,000 mg/kg	10 mg/kg	---	---	0.012 mg/kg	0.067 mg/kg
1-Methylnaphthalene	90-12-0	5,300 mg/kg	10 mg/kg	---	---	0.014 mg/kg	0.067 mg/kg
2-Methylnaphthalene	91-57-6	6,600 mg/kg	10 mg/kg	---	---	0.016 mg/kg	0.067 mg/kg

Notes:
[1]Analytical MDLs and QLs are those documented in validated methods.
[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

CAS – Chemical Abstract Service
MDL – Method Detection Limit
Mg/kg – Milligram per kilogram
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
QL – Quantitation Limit

QAPP Worksheet #16
Project Schedule/Timeline Table

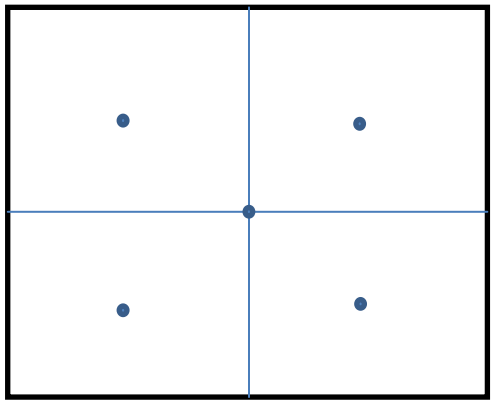
Activities	Organization	Dates (Month Day, Year)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
QAPP Preparation	WESTON	September 23, 2013	September 27, 2013	QAPP	October 1, 2013 (Draft)
Excavation and Confirmation Sampling	ERRS/WESTON	September 23, 2013	October 31, 2013	Confirmation samples to laboratory	TBD
Laboratory Analysis	Test America, Inc.	September 23, 2013	October 18, 2013	Laboratory Data Report	TBD
Final Project Report	WESTON	October 1, 2013	November 8, 2013	Final Report	TBD

Notes:
ERRS – Emergency and Rapid Response Services
QAPP – Quality Assurance Project Plan
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #17
Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (*e.g.*, grid system, biased statistical approach): The selected removal action for the Site consists of excavation of lead contaminated soil to below 800 mg/kg lead, 15,000 mg/kg vanadium, 1,300 mg/kg benzene, and varies for PAHs (see Worksheet #15). Specifically, two areas were found to be contaminated; one area is in the southwest corner of the site and the other is located on the northwest corner of the site. Once coal tar solids and impacted soil has been removed from an area, confirmation soil samples will be collected for off-site laboratory analysis as discussed below.

It is estimated that approximately 15 soil samples will be collected to confirm that cleanup goals were reached during excavation. At a minimum, one composite sample will be collected for each 400 square foot (ft^2) area excavated. The samples will be collected from the excavated surface at a depth of approximately 0 to 6 feet below ground surface (bgs). The composite sample will be a five point composite consisting of one sample from the center of the 400-ft^2 area and 4 samples from the center of each quadrant of the 400-ft^2 area. The diagram below illustrates how this will be accomplished; the dots are the 5 sample points that will make up the composite sample. These 5 samples will then be homogenized into one composite sample.



Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [Refer to Worksheet #18 for details]:
Surface soil (0-6 feet bgs) from the excavation areas (confirmation soil samples) will be sampled. The confirmation soil samples will be analyzed for total lead, vanadium, and benzene; the concentration levels are expected to be low to high. One field duplicate will be collected for

every 10 samples collected. The number of confirmation soil samples will be dependent on the size of the excavations and amount of soil excavated. It is expected that approximately 15 confirmation soil samples will be collected. Samples will be collected as excavation areas are completed.

Notes:
bgs – below ground surface
COC – Chain-of-Custody
ERRS – Emergency and Rapid Response Services
mg/kg – Milligram per kilogram
PAH – Polynuclear Aromatic Hydrocarbon
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
START – Superfund Technical Assessment and Response Team
WESTON – Weston Solutions, Inc.

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ ID Number	Matrix	Depth	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)[1]	Sampling SOP Reference[2]	Rationale for Sampling Location
Excavation: SIR-SS01-mmddyy (up to S15)	Soil	0-6 feet at the surface of the completed excavation	Lead, Vanadium, Benzene, and PAHs	High/Medium/ Low	Up to 15 samples plus two field duplicates	See Worksheet #17	See Worksheet #17

Notes:
[1]MS/MSD and field duplicate samples will be collected at a frequency of 1 for every 20 samples.
[2]Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

ID – Identification
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
SOP– Standard Operating Procedure

QAPP Worksheet #19
Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[1]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Soil	Lead and Vanadium	High/Medium/Low	LSOP-1	4 oz	1 4-oz wide-mouth glass jar	Cool to 4ºC	180 days from collection to analysis
Soil	Benzene	High/Medium/Low	LSOP-2	15 g	(3) EnCoreTM 5g samplers	Cool to 4ºC	48 hours
Soil	PAHs	High/Medium/Low	LSOP-3	4 oz	1 4-oz wide-mouth glass jar	Cool to 4ºC	14 days from collection to analysis

Notes:
Worksheet #19 content to be verified when ERRS selects and awards the analytical laboratory.
[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

ºC – Degrees Celsius
g – gram
oz – ounce
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure

QAPP Worksheet #20
Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equip. Blanks	Total No. of Samples to Lab
Soil	Lead and Vanadium	High/Medium/Low	LSOP-1	15	2	1	0	0	17
	Benzene	High/Medium/Low	LSOP-2	15	2	1	0	0	17
	PAHs	High/Medium/Low	LSOP-3	15	2	1	0	0	17

Notes:
[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
[2]If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

MS – Matrix Spike
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure

QAPP Worksheet #21
Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
NA	NA (see Worksheet #17)				

Notes:

NA – Not Applicable

QAPP Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
UltraRAE	At least once per day of use.	Charge batteries daily	Air monitoring	Frequent field check to ensure that the instrument particulate filter is free of particulate/debris	Frequently (at least once daily)	Tip is free of particulate/debris	Replace Filter	WESTON START Site Leader	Manufacturer's Instructions
AreaRAEs	At least once per day of use.	Charge batteries daily	Air monitoring	Frequent field check to ensure that the instrument particulate filters are free of particulate/debris	Frequently (at least once daily)	Tip is free of particulate/debris	Replace Filter	WESTON START Site Leader	Manufacturer's Instructions

Notes:
[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP Worksheet #23
Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
LSOP-1	SOP ID: 6010 / NV06-44.14a; Method 6010B/C: Inductively Coupled Plasma-Atomic Emission Spectrometry	Definitive	Total Lead and Vanadium	ICP	Test America, Inc.	N
LSOP-2	SOP ID: 8260 / NV05-77, Rev. 18; Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry; SW-846 METHOD 8260B/C	Definitive	Benzene	GC/MS	Test America, Inc.	N
LSOP-3	SOP ID: 8270 / NV/SA04-22.15a; Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry; Method 8270C/D	Definitive	PAHs	GC/MS	Test America, Inc.	N

Notes:
GC/MS – Gas Chromatography/ Mass Spectrometry
QAPP – Quality Assurance Project Plan
ICP – Inductively Coupled Plasma Spectrophotometer
LSOP – Laboratory Standard Operating Procedure
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure
WESTON – Weston Solutions, Inc.

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP	See referenced SOP in Appendix A				Test America, Inc. Chemist	LSOP-1
GC/MS	See referenced SOP in Appendix A				Test America, Inc. Chemist	LSOP-2, LSOP-3

Notes:
[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

GC/MS – Gas Chromatography/ Mass Spectrometry
ICP – Inductively Coupled Plasma Instrument
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
ICP	See referenced SOP in Appendix A						Test America, Inc. Chemist	LSOP-1
GC/MS	See referenced SOP in Appendix A						Test America, Inc. Chemist	LSOP-2, LSOP-3

Notes:
[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

GC/MS – Gas Chromatography/ Mass Spectrometry
ICP – Inductively Coupled Plasma Spectrometry
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure

QAPP Worksheet #26
Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
• Sample Collection (Personnel/Organization): WESTON START Site Lead or ERRS designated personnel
• Sample Packaging (Personnel/Organization): WESTON START Site Lead or ERRS designated personnel
• Coordination of Shipment (Personnel/Organization): ERRS Laboratory Procurement Coordinator or WESTON START Lead
• Type of Shipment/Carrier: Federal Express, delivery, or courier pick-up
SAMPLE RECEIPT AND ANALYSIS
• Sample Receipt (Personnel/Organization): Laboratory Sample Login
• Sample Custody and Storage (Personnel/Organization): Laboratory Sample Receipt
• Sample Preparation (Personnel/Organization): Laboratory Personnel
• Sample Determinative Analysis (Personnel/Organization): Laboratory Personnel
SAMPLE ARCHIVING
• Field Sample Storage (No. of days from sample collection): All samples will be sent to the laboratory. The laboratory shall retain the samples in accordance with their laboratory SOPs.
• Sample Extract/Digestate Storage (No. of days from extraction/digestion): Six months or per the labs policy.
• Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
• Personnel/Organization: Laboratory
• Number of Days from Analysis: In accordance with the laboratory SOPs.

Notes:
ERRS – Emergency and Rapid Response Services
SOPs – Standard Operating Procedures
START – Superfund Technical Assessment and Response Team
WESTON – Weston Solutions, Inc.

QAPP Worksheet #27
Sample Custody Requirements

Chain-of-Custody Procedures: A COC record will be maintained from the time the sample is collected until its delivery to the laboratory. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC record will be filled out for each sample at each sampling location. Each individual in possession of the samples must sign and date the sample COC document. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time, will be documented. A copy of the COC is retained by the site leader for the site file. When samples (or groups of samples) are not under direct control of the individual responsible for them, they must be stored in a locked container sealed with a custody seal. The COC record will be considered completed upon receipt at the laboratory. The COC record should include (at minimum) the following:

- Type (s) of analysis(es) to be performed
- Sample ID number
- Sample information
- Sample station location
- Sample date
- Name(s) and signature(s) of sampler(s)
- Signature(s) of any individual(s) with control over samples

A separate COC form must accompany each cooler in each shipment. Within the laboratory, the person responsible for sample receipt must sign and date the COC form; verify that custody seals are intact on shipping containers; compare samples received against those listed on the COC form; examine all samples for possible shipping damage, leakage, and improper sample preservation; note on the COC record or laboratory receiving documentation that specific samples were damaged; notify sampling personnel as soon as possible so that appropriate samples may be resampled; verify that sample holding times have not been exceeded; maintain laboratory COC documentation; and place the samples in appropriate laboratory storage. If requested, the laboratory may submit internal COC documentation with the data package. Final sample disposition is completed according to laboratory license requirements.

Sample Identification Procedures: All samples for laboratory analysis, including QC samples, will be given a unique sample number. The sample numbers will be recorded in the field logbook, the COC paperwork, and the shipment documents. The sample number highlights the suspected contaminated area and location, and will be used for documentation purposes in field logbooks, as well as for presentation of the analytical data in memoranda and reports. The confirmation sample numbering system will be composed of the components below.

SIR-SSXX-mmddyy

Where:

- "SIR" indicates that the sample is from the Springfield Iron Removal Action Site
- "SS" indicates that the sample is a site soil sample
- "XX" indicates the sequential sampling location (01, 02, etc)
- "mmddyy" indicates the date sampled

Field duplicate samples will be designated with a "D" suffix. An example of the sample identifications for the Site is as follows:

- SIR-SS01-092313: Soil confirmation sample collected at location 01, on September 23, 2013.

Notes:
COC – Chain-of-Custody
DUP – Duplicate
ID - Identification
MS/MSD – Matrix spike/matrix spike duplicate
QC – Quality Control

QAPP Worksheet #28A
QC Samples Table

		Concentration Level:	High/Medium/Low
Analytical Group:	Lead, Vanadium	Sampler's Name:	TBD
Analytical Method/ SOP Reference:	LSOP-1	Field Sampling Organization:	WESTON or ERRS
Matrix:	Soil	Analytical Organization:	Test America, Inc.
Sampling SOP:	See Worksheet #17	No. of Sample Locations:	Up to 15

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Laboratory Duplicate	1	RPD ≤ 20%	Flag associated data as estimated	Chemist	Laboratory Precision	See LSOP-1
Method Blank	1	No target analyte concentrations above reporting limit	Flag data at less than 10 times the blank concentration as not detected	Chemist	Laboratory Contamination	
LCS	1	80 to 120 % Recovery	Flag associated data as estimated	Chemist	Laboratory Accuracy	
MS/MSD	1	75 to 125 % Recovery	Flag associated data as estimated	Chemist	Matrix Interference/ Laboratory Accuracy	

Notes:
DQI – Data Quality Indicator
ERRS – Emergency and Rapid Response Services
QAPP – Quality Assurance Project Plan
QC – Quality Control
LCS/LCSD – Laboratory Control Sample/Laboratory Control Sample Duplicate
MS/MSD – Matrix Spike/Matrix Spike Duplicate
RL – Reporting Limit
RPD – Relative Percent Difference
SOP – Standard Operation Procedure
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #28B
QC Samples Table

		Concentration Level:	High/Medium/Low
Analytical Group:	Benzene	Sampler's Name:	TBD
Analytical Method/ SOP Reference:	LSOP-2	Field Sampling Organization:	WESTON or ERRS
Matrix:	Soil	Analytical Organization:	Test America, Inc.
Sampling SOP:	See Worksheet #17	No. of Sample Locations:	Up to 15

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Laboratory Duplicate	1	RPD ≤ 20%	Flag associated data as estimated	Chemist	Laboratory Precision	See LSOP-2
Method Blank	1	No target analyte concentrations above reporting limit	Flag data at less than 10 times the blank concentration as not detected	Chemist	Laboratory Contamination	
LCS	1	70 to 130 % Recovery	Flag associated data as estimated	Chemist	Laboratory Accuracy	
MS/MSD	1	70 to 130 % Recovery	Flag associated data as estimated	Chemist	Matrix Interference/ Laboratory Accuracy	

Notes:
DQI – Data Quality Indicator
ERRS – Emergency and Rapid Response Services
QAPP – Quality Assurance Project Plan
QC – Quality Control
LCS – Laboratory Control Sample
WESTON – Weston Solutions, Inc.
MS/MSD – Matrix Spike/Matrix Spike Duplicate
RL – Reporting Limit
RPD – Relative Percent Difference
SOP – Standard Operation Procedure
TBD – To Be Determined

QAPP Worksheet #28C
QC Samples Table

		Concentration Level:	High/Medium/Low
Analytical Group:	PAHs	Sampler's Name:	TBD
Analytical Method/ SOP Reference:	LSOP-3	Field Sampling Organization:	WESTON or ERRS
Matrix:	Soil	Analytical Organization:	Test America, Inc.
Sampling SOP:	See Worksheet #17	No. of Sample Locations:	Up to 15

QC Sample	Frequency / Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Laboratory Duplicate	1	RPD ≤ 20%	Flag associated data as estimated	Chemist	Laboratory Precision	See LSOP-3
Method Blank	1	No target analyte concentrations above reporting limit	Flag data at less than 10 times the blank concentration as not detected	Chemist	Laboratory Contamination	
LCS	1	70 to 130 % Recovery	Flag associated data as estimated	Chemist	Laboratory Accuracy	
MS/MSD	1	70 to 130 % Recovery	Flag associated data as estimated	Chemist	Matrix Interference/ Laboratory Accuracy	

Notes
DQI – Data Quality Indicator
ERRS – Emergency and Rapid Response Services
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
QC – Quality Control
LCS – Laboratory Control Sample
MS/MSD – Matrix Spike/Matrix Spike Duplicate
RL – Reporting Limit
RPD – Relative Percent Difference
SOP – Standard Operation Procedure
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #29
Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Logbook(s)	Logbook(s)	Sample Receipt, Custody, and Tracking Records	Corrective Action Reports	Investigation Summary Report
COC Forms	Final Analytical Data Summary Report	Preliminary analytical data reports		
Photos		Final Analytical Data Summary Reports		
Air bills		Laboratory Electronic Data Deliverables		
GPS Coordinates		Sample Preparation Logs		
		Run Logs		
		Equipment Maintenance, Testing, and Inspection Logs		
		Instrument printouts (raw data)		
		Quality Control Sample Summary Forms		
		Sample Disposal Records		
		Corrective Action Reports		

Notes:
COC – Chain-of-Custody
QAPP – Quality Assurance Project Plan

QAPP Worksheet #30
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Soil	Total Lead, Vanadium, Benzene, and PAHs	High/Medium/Low	Excavation Areas, SIR-SSXX-mmddyy	LSOP-1, LSOP-2, LSOP-3	TBD	Test America, Inc., 2960 Foster Creighton Drive Nashville, TN 37204 (615) 301-5757 - Office Ryan.fitzwater@testamericainc.com	TBD

Notes:
ID – Identification
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure
TBD – To Be Determined

QAPP Worksheet #31
Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing CA (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Field Audit	In accordance with EPA OSC Request	Internal	Weston Solutions, Inc.	Tonya Balla or her designee, Project Manager, WESTON START	TBD	TBD	TBD

Notes:
CA – Corrective Action
QAPP – Quality Assurance Project Plan
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #32
Assessment Findings and Response Actions

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Field Audit	Written memo	TBD	One day	Obtain documentation of corrective action from Field Team Members	Tonya Balla, Project Manager, WESTON START	Two days

Notes:
QAPP – Quality Assurance Project Plan
QC – Quality Control
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #33
QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Final Project Report	To be prepared upon completion of removal activities at the Site	One month following completion of removal activities at the Site	TBD	Jaime Brown, OSC, EPA Region V
Monthly Report	Every month for the prior month activities	20th of month for the prior month activities	Tonya Balla, PM, WESTON	Jaime Brown, OSC, EPA Region V

Notes:
ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
TBD – To Be Determined
WESTON – Weston Solutions, Inc.

QAPP Worksheet #34
Sampling and Analysis Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
COC Forms	The Site Leader will submit COC forms to the ERRS RM within 2 hours following all sample shipments to the laboratory. The RM will review the COC forms for completeness to ensure that the proper analyses are being performed.	Internal	Toby Viehweg, ERRS RM
Logbook	The PM will review the logbook for accuracy and completeness following field sampling activities.	Internal	Tonya Balla, WESTON START
Laboratory Data	All laboratory data will be verified by the QA officer of the laboratory performing the sample analyses. The WESTON QA Officer will perform a compliance check of all data received from the laboratory.	External Internal	QA Officer, Laboratory WESTON QA Officer

Notes:
COC – Chain-of-Custody
ERRS – Emergency and Rapid Response Services
PM – Project Manager
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
RM – Response Manager
START – Superfund Technical Assessment and Response Team
TBD – To Be Determined
WESTON – Weston Solutions, Inc

QAPP Worksheet #35
Sampling and Analysis Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	SOPs and logbook	The PM will ensure that all SOPs were followed in the field through daily conversations with the site leader and review of the site logbook.	Tonya Balla, START
IIb	Preliminary Data and Final Analytical Data Package	The ERRS SMC will review the preliminary data and final analytical data package to ensure that all analyses requested were received and to ensure that required project quantitation limits were met. The START QA Officer or their designee will perform a compliance check of all data received from the laboratory.	TBD, ERRS

Notes:
ERRS – Emergency and Rapid Response Services
PM – Project Manager
QAPP – Quality Assurance Project Plan
QC – Quality Control
SMC – Sample Management Coordinator
SOP – Standard Operating Procedure
START – Superfund Technical Assessment and Response Team
TBD – To Be Determined

QAPP Worksheet #36
Sampling and Analysis Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIb	Soil	Total Lead, Vanadium, Benzene, and PAHs	High/Medium/Low	EPA CLP NFG for Inorganic Data	START

Notes:
ERRS – Emergency and Rapid Response Services
NA – Not Applicable.
PAH – Polynuclear Aromatic Hydrocarbon
QAPP – Quality Assurance Project Plan
START – Superfund Technical Assessment and Response Team
TBD – To Be Determined
EPA – United States Environmental Protection Agency
WESTON – Weston Solutions, Inc.

QAPP Worksheet #37
Data Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: Data, generated by the laboratory, are tabulated and reviewed for precision, accuracy, representativeness, and completeness by the site leader for field data or by the sample management coordinator for laboratory data from a fixed laboratory. The review of these DQI will compare the DQI with the DQO detailed in the project-specific QAPP and in the analytical methods used.

Questions about data, as observed during the data review process, are resolved by contacting the respective site personnel and laboratories for resolution. All communications are documented including the resolution to the observed deficiencies. Hard copies of all original data and deliverables are kept in the TDD file.

When the data do not meet the project DQOs, WESTON START will investigate the root cause to the deficiency. Reasons may include laboratory operation, such as the laboratory's failure to adjust the extraction weight on high-moisture-content soil, failure of laboratory reporting limits to meet site Action Limits, or poor correlation between field screening and laboratory results. In these situations, WESTON START will discuss corrective actions with the EPA OSC. These actions may include:

- Resampling for all or some of the parameters
- Preparing a technical memorandum to the site file, detailing limitations to the data
- Validating the data at a higher tier level to better qualify the results
- Preparing a technical memorandum determining the bias of field results

Describe the evaluative procedures used to assess overall measurement error associated with the project: The following specific items will be assessed in the manner described below:

Precision – Results of all laboratory duplicates and field duplicates will be presented in the laboratory data validation report. For each duplicate pair, the RPD will be calculated for each analyte with results greater than or equal to the quantitation limit. The RPDs will be checked against the measurement performance criteria presented on Worksheet #12. The RPDs exceeding criteria will be identified on the tables in the final report with appropriate qualifiers. A discussion will follow summarizing the results of the laboratory precision. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described in the final report.

Accuracy/Bias Contamination – Results for all laboratory method blanks and instrument blanks will be presented in the laboratory data validation report. The results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12.

Results for analytes that exceed criteria will be identified on the tables in the final report with appropriate qualifiers. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Overall Accuracy/Bias – The results for the continuing calibration standards will be presented in the laboratory data validation report. These results will be compared to the requirements listed on Worksheet #12. A discussion will follow summarizing overall accuracy/bias. Any conclusions about the overall accuracy/bias of the analyses will be drawn and any limitations on the use of the data will be described.

Sensitivity – All sample results will be presented in tabular format for each analyte. The sample results for each analyte will be checked against the method detection limits. Results for analytes that do not meet the contract required quantitation limits will be discussed. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.

Representativeness – Representativeness will be maintained by the site leader who will ensure that all sampling personnel are adhering to the sampling procedures dictated in the field sampling plan. In addition, the PM will be in close contact with the Site Leader to ensure that proper sampling techniques are being followed. Any conclusions about the representativeness of the sampling will be drawn and any limitations on the use of the data will be described.

Completeness – A completeness check will be done on all samples collected in the field and data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated as follows. For each sample collected, completeness will be calculated as the number of samples collected and number of analyses performed, divided by the total number of planned sample collection points and analyses. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

Reconciliation – Each of the project quality objectives presented on Worksheet #12 will be examined to determine if the objective was met. Each analysis will first be evaluated in terms of the major impacts observed from the data validation, DQIs, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the usability of the data from all analyses for an objective, it will be determined if the project quality objective was met. The final report will include a summary of all the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

Identify the personnel responsible for performing the usability assessment: The site leader will determine the usability of field data. The ERRS SMC and WESTON QA Officer will do a compliance check of the data to determine the usability of analytical data. The PM, Tonya

Balla, will be responsible for the overall usability to meet project objectives.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: Overall usability of data to meet project objectives will be described in the final report to be prepared by the PM.

Notes:
DQI – Data Quality Indicator
DQO – Data Quality Objective
ERRS – Emergency and Rapid Response Services
OSC – On-Scene Coordinator
PM – Project Manager
QAPP – Quality Assurance Project Plan
RPD – Relative Percent Difference
SMC – Sample Management Coordinator
START – Superfund technical Assessment and Response Team
TDD – Technical Direction Document
WESTON – Weston Solutions, Inc.

APPENDIX A

LABORATORY SOPs

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **8270 / NV/SA04-22.15a** Effective Date: **8/6/2013**

Last Mod. Date: **3/29/2013**

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Section 16.0, Modifications; Section 17.0, Attachments

CONTROLLED DISTRIBUTION
ISSUED TO: QA Server, 04B

Revision Number with Mod ID: **15b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Add bold text, delete crossed-out text.

Section 16.0, Method Modification: Add a last sentence to item 4:

Item	Modification
4	SIM is not allowed for South Carolina samples unless pre-approved by the state on a project-specific basis. **SC has not approved RVE/LVI.**

Section 17.0, Attachments, Attachment 1, Characteristic Ions for Semivolatile Compounds: Modify the characteristic ions for the following compounds:

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
o,o,o-Triethylphosphorthioate	5.302	198	**121, 97** ~~80, 53, 54164, 63~~
1,4-Phenylenediamine	5.734	**108** ~~198~~	80, **107** ~~53, 54, 52~~
n-Octadecane	7.586	**57** ~~58~~	71~~, 85~~
Hexachlorophene	9.070	**196** ~~185~~	**198, 209** ~~209, 406~~

Michael A. Brum 8/6/13	8/6/13
Technical and Quality Assurance Approval Date	Operations Manager Approval Date

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **8270 / NV/SA04-22.15** Effective Date: **3/29/2013**

Last Mod. Date: 12/31/12

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Section 3.0, Definitions; Section 7.0, Reagents and Standards, Section 9.1, Sample QC, Section 10.2, Calibration, Section 16.0, Method Modification

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 04B

Revision Number with Mod ID: **15a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Add bold text, delete crossed-out text.

Section 3.0, Definitions: Add a last sentence to 3.1 Reduced Volume Extraction / Large Volume Injection (RVE/LVI)**: Generally, reduce all concentrations by a factor of RVE/LVI, i. e., 5.**

Section 7.0, Reagents and Standards

7.6 GC/MS Tuning Standard: Add to the first sentence: "A Methylene chloride solution containing 50 µg/mL **[RVE/LVI: 10 µg/L]** of Decafluorotriphenylphosphine (DFTPP) is prepared.

7.7 Surrogate Standards: To the bullet item, add a last sentence: **Dilute by five for RVE/LVI.**

Section 9.1, Sample QC, Surrogate recoveries: Delete the phrase: The limits for surrogate recoveries are updated biannually ~~(see TestAmerica Nashville's current Control Limits Manual (CLM))~~.

Section 10.2, Calibration, Initial Calibration, Steps 1 and 2: Add bold column.

- Prepare calibration standards at five (minimum) different concentrations.

Traditional Volume Concentration (µg/mL)	**RVE/LVI Concentration (µg/mL)**	µL of 200 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL of 200 µg/mL standard/500 µL (5 µL injection)
2	**0.4**	5	1

10	**2**	25	5
20	**4**	50	10
50	**10**	125	25
80	**16**	200	40
100	**20**	250	50

For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:

Concentration (µg/mL)	**RVE/LVI Concentration (µg/mL)**	µL of 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL of 10 µg/mL standard/500 µL(5 µL injection)
0.05*	**0.01**	2.5	0.5
0.1	**0.02**	5	1
0.5	**0.1**	25	5
1	**0.2**	50	10
5	**1.0**	250	50
10	**2.0**	500	100

*The lowest calibration ~~0.05 µg/mL~~ standard must be used for low-level SIM analysis on samples from Wisconsin.

Section 16.0, Method Modification: Add item 5 to the table:

Item	Modification
5	**RVE/LVI**

Michael A. Brown 3/11/13	3/11/13
Technical and Quality Assurance Approval Date	Operations Manager Approval Date

SOP Number/Revision No.: **8270 / NV/SA04-22.15** Effective Date: **3/29/2013**

Last Mod. Date: 12/31/12

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Revision Number with Mod ID: **15a**

Title: SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHOD 8270C/D

Approvals (Signature/Date)			
	12/28/12		12/28/12
Cory Spry Extractables Operations Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date
	12/28/12		
Michael H. Dunn Technical Director Quality Assurance Manager	Date		

Analyze and report by 8270D for Canadian, **NJ, NC, OK, SC, and WV** samples.

1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of oily wastes, soils/sediments, concrete, and water samples. The following compounds can be determined by this method:

Analyte	CAS #	Analyte	CAS #
Acenaphthene[1, 2, 5]	83-32-9	Hexachlorocyclopentadiene[1, 2]	77-47-4
Acenaphthene-d_{10} (IS)		Hexachloroethane[1, 2]	67-72-1
Acenaphthylene[1, 2, 5]	208-96-8	Hexachlorophene[2]	70-30-4
Acetophenone[2]	98-86-2	Hexachloropropene[2]	1888-71-7
2-Acetylaminofluorene[2]	53-96-3	Indeno(1,2,3-cd)pyrene[1, 2, 5]	193-39-5
4-Aminobiphenyl[2]	92-67-1	*Indene*[4]	
Aniline[2]	62-53-3	Isodrin[2]	465-73-6
Anthracene[1, 2, 4, 5]	120-12-7	Isophorone[1, 2]	78-59-1
Aramite[2]	140-57-8	*cis-Isosafrole*[2]	17627-76-8
Azobenzene[3]	103-33-3	*trans-Isosafrole*[2]	4043-71-4
Benzidine[3]	92-87-5	Kepone[2]	143-50-0
Benzoic acid[3]	65-85-0	Methapyrilene[2]	91-80-5
Benz(a)anthracene[1, 2, 4, 5]	56-55-3	3-Methylcholanthrene[2]	56-49-5
Benzo(b)fluoranthene[1, 2, 4, 5]	205-99-2	*6-Methylchrysene*[4]	1705-85-7
Benzo(j)fluoranthene[4]		4,4'-Methylenebis(2-chloroaniline) (MOCA)[3]	101-14-4
Benzo(k)fluoranthene[1, 2, 4, 5]	207-08-9	Methyl methanesulfonate[2]	66-27-3
Benzo(g,h,i)perylene[1, 2, 5]	191-24-2	*2-Methylnaphthalene*[3, 4, 5]	90-12-0
Benzo(a)pyrene[1, 2, 4, 5]	50-32-8	2-Methylnaphthalene[1, 2, 5]	91-57-6
Benzyl alcohol[2]	100-51-6	Methyl parathion[2]	298-00-0
Bis(2-chloroethoxy)methane[1, 2]	111-91-1	2-Methylphenol[1, 2, 4]	95-48-7
Bis(2-chloroethyl)ether[1, 2]	111-44-4	3-Methylphenol[1, 2, 4]	108-39-4
Bis(2-chloroisopropyl)ether[1, 2]	108-60-1	4-Methylphenol[1, 2, 4]	106-44-5
Bis(2-ethylhexyl)adipate[3]	103-23-1	Naphthalene[1, 2, 4, 5]	91-20-3
Bis(2-ethylhexyl)phthalate[1, 2, 4]	117-81-7	Naphthalene-d_8 (IS)	
Bisphenol A[3]	80-05-7	1,4-Naphthoquinone[2]	130-15-4
4-Bromophenyl phenylether[1, 2]	101-55-3	1-Naphthylamine[2]	134-32-7
Butyl benzyl phthalate[1, 2, 4]	85-68-7	2-Naphthylamine[2]	91-59-8
Carbazole[1]	86-74-8	2-Nitroaniline[1, 2]	88-74-4
4-Chloroaniline[1, 2]	106-47-8	3-Nitroaniline[1, 2]	99-09-2
Chlorobenzilate[2]	510-15-6	4-Nitroaniline[1, 2]	100-01-6
4-Chloro-3-methylphenol[1, 2]	59-50-7	Nitrobenzene[1, 2]	98-95-3
1-Chloronaphthalene[3]	90-13-1	Nitrobenzene-d_5 (surr)	
2-Chloronaphthalene[1, 2]	91-58-7	2-Nitrophenol[1]	88-75-5
2-Chlorophenol[1, 2]	95-57-8	4-Nitrophenol[1, 2]	100-02-7
2-Chlorophenol-d_4 (surr)		5-Nitro-o-toluidine[2]	99-55-8
4-Chlorophenyl phenylether[2]	7005-72-3	Nitroquinoline-1-oxide[2]	56-57-5
Chrysene[1, 2, 4, 5]	218-01-9	n-Nitrosodi-n-butylamine[2]	924-16-3
Chrysene-d_{12} (IS)		n-Nitrosodiethylamine[2]	55-18-5
n-Decane[3]	124-18-5	n-Nitrosodimethylamine[2]	62-75-9
Diallate (cis and trans)[2]	2303-16-4	n-Nitrosomethylethylamine[2]	10595-95-6
Dibenz(a,h)acridine[4]	226-36-8	n-Nitrosodiphenylamine[1, 2] and Diphenylamine	86-30-6 and 122-39-4
Dibenz(a,j)acridine[3]	224-42-0	n-Nitrosodi-n-propylamine[1, 2]	621-64-7
Dibenz(a,h)anthracene[1, 2, 4, 5]	53-70-3	n-Nitrosomorpholine[2]	59-89-2
Dibenzofuran[1, 2]	132-64-9	n-Nitrosopiperidine[2]	100-75-4
2,3-Dichloroaniline[3]	608-27-5	n-Nitrosopyrrolidine[2]	930-55-2

1,2-Dichlorobenzene[1, 2, 4]	955-50-1	*n-Octadecane*[3]	593-45-3
1,2-Dichlorobenzene-d_4(surr)		Parathion[2]	56-38-2
1,3-Dichlorobenzene[1, 2, 4]	541-73-1	Pentachlorobenzene[2]	608-93-5
1,4-Dichlorobenzene[1, 2, 4]	106-46-7	*Pentachloroethane*[2]	76-01-7
1,4-Dichlorobenzene-d_4(IS)		Pentachloronitrobenzene[2]	82-68-8
3,3'-Dichlorobenzidine[1, 2]	91-94-1	Pentachlorophenol[1, 2]	87-86-5
2,4-Dichlorophenol[1, 2]	120-83-2	Perylene-d_{12} (IS)	
2,6-Dichlorophenol[2]	87-65-0	Phenacetin[2]	62-44-2
Diethyl phthalate[1, 2, 4]	84-66-2	Phenanthrene[1, 2, 4, 5]	85-01-8
Dimethoate[2]	60-51-5	Phenanthrene-d_{10} (IS)	
Dimethylaminoazobenzene[2]	60-11-7	Phenol[1, 2, 4]	108-95-2
7,12-Dimethylbenz(a)anthracene[2, 4]	57-97-6	Phenol-d_5 (surr)	
3,3'-Dimethylbenzidine[2]	119-93-7	1,4-Phenylenediamine[3]	106-50-3
2,4-Dimethylphenol[1, 2, 4]	105-67-9	Phorate[2]	298-02-2
a,a- Dimethylphenethylamine[2]	122-09-8	2-Picoline (2-Methylpyridine)[2]	109-06-8
Dimethyl phthalate[1, 2, 4]	131-11-3	Pronamide[2]	23950-58-5
Di-n-butyl phthalate[1, 2, 4]	84-74-2	Pyrene[1, 2, 4, 5]	129-00-0
1,3-Dinitrobenzene[2]	99-65-0	Pyridine[2, 4]	110-86-1
4,6-Dinitro-2-methylphenol[1, 2]	534-52-1	*Quinoline*[3]	91-22-5
2,4-Dinitrophenol[1, 2, 4]	51-28-5	Safrol[3]	94-59-7
2,4-Dinitrotoluene[1, 2, 5]	121-14-2	Terphenyl-d_{14}(surr)	1718-51-0
2,6-Dinitrotoluene[1, 2, 5]	606-20-2	*Alpha-Terpineol*[3]	7785-53-7
Dinoseb[2]	88-85-7	1,2,4,5-Tetrachlorobenzene[2]	95-94-3
1,4-Dioxane	123-91-9	2,3,4,6-Tetrachlorophenol[2]	58-90-2
1,2-Diphenylhydrazine[3]	122-66-7	*Tetraethyl dithiopyrophosphate (Sulfotepp)*[2]	3689-24-5
Di-n-octyl phthalate[1, 2, 4]	117-84-0	Tetraethylpyrophosphate[3]	107-49-3
Disulfoton[2]	298-04-4	Thionazine[2]	297-97-2
Ethyl methanesulfonate[2]	62-50-0	Thiophenol[4]	108-98-5
Famphur[3]	52-85-7	o-Toluidine[2]	95-53-4
Fluoranthene[1, 2, 4, 5]	206-44-0	2,4,6-Tribromophenol (surr)	118-79-6
Fluorene[1, 2, 5]	86-73-7	1,2,4-Trichlorobenzene[1, 2]	120-82-1
2-Fluorobiphenyl(surr)	321-60-8	2,4,5-Trichlorophenol[1, 2]	95-95-4
2-Fluorophenol (surr)	367-12-4	2,4,6-Trichlorophenol[1, 2]	88-06-2
Hexachlorobenzene[1, 2]	118-74-1	o,o,o-Triethylphosphorothioate[2]	126-68-1
Hexachlorobutadiene[1]	87-68-3	1,3,5-Trinitrobenzene[2]	99-35-4
Compounds in italics are not present in the EPA method.			
[1] – Normal laboratory 8270 compounds. ***For OH VAP projects, report only compounds designated by a 1 superscript; see Attachment 5.***			
[2] - Appendix IX compounds (by request only)			
[3] - additional compounds available by this method (by request only)			
[4] - Skinner List for Refinery Waste compounds (by request only)			
[5] - Compounds that are available by GC/MS-SIM (by request only)			
IS = These compounds are used as internal standards.			
surr = These compounds are used as surrogates.			

This method is used to quantitate neutral, acidic, and basic organic compounds that are soluble in Methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica capillary column coated with a slightly polar methyl silicone phase. This method is not appropriate for the quantitation of multi-component analytes, e. g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analyses. This method is appropriate for confirmation of the presence of these analytes when concentration in the extract

permits. However, it is appropriate for the multi-component analyte, Diesel Range Organics (DRO), as requested by Missouri and California; see Attachment 6.

1.2 Reporting Limits: The laboratory typical report limit (RL) is approximately 2 - 100 µg/L for water samples, 67 - 670 µg/kg (wet weight) for soil/sediment samples, and 10 - 1000 mg/kg for wastes (dependent on matrix and method of preparation). See the following table for typical RLs for each compound. For the most current analyte RLs, refer to LIMS.

Typical Reporting Limits for 8270 Compounds

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**Acenaphthene**	10	0.333	♦*Isosafrole*	50	1.67
♦**Acenaphthylene**	10	0.333	♦*Kepone*	10	0.333
♦*Acetophenone*	10	0.333	♦*Methapyrilene*	50	0.333
♦*2-Acetylaminofluorene*	10	0.333	♦*3-Methylcholanthrene*	10	0.333
♦*4-Aminobiphenyl*	10	0.333	♣*6-Methylchrysene*	10	0.333
♦*Aniline*	10	0.333	♦*Methyl methanesulfonate*	10	0.333
♦♣**Anthracene**	10	0.333	♣*1-Methylnaphthalene*	10	0.333
♦*Aramite*	50	1.67	♦**2-Methylnaphthalene**	10	0.333
Atrazine	10	0.333	♦*Methyl parathion*	10	1.67
Azobenzene	10	0.333	♦♣**2-Methylphenol**	10	0.333
Benzaldehyde	10	1.67	♦♣**3,4-Methylphenol**	10	0.333
Benzidine	100	1.67	♦♣**Naphthalene**	10	0.333
Benzoic acid	50	1.67	♦*1,4-Naphthoquinone*	10	1.67
♦♣**Benzo(a)anthracene**	10	0.333	♦*1-Naphthylamine*	10	0.333
♦♣**Benzo(a)pyrene**	10	0.333	♦*2-Naphthylamine*	10	0.333
♦♣**Benzo(b)fluoranthene**	10	0.333	♦**2-Nitroaniline**	25	0.833
♦**Benzo(g,h,i)perylene**	10	0.333	♦**3-Nitroaniline**	25	0.833
♣*Benzo(j)fluoranthene*	10	0.333	♦**4-Nitroaniline**	25	0.833
♦♣**Benzo(k)fluoranthene**	10	0.333	♦**Nitrobenzene**	10	0.333
♦*Benzyl alcohol*	10	0.333	♦*5-Nitro-o-toluidine*	10	1.67
Biphenyl	10	0.333	♦**2-Nitrophenol**	10	0.333
♦**Bis(2-chloroethoxy) methane**	10	0.333	♦♣**4-Nitrophenol**	25	0.833
♦**Bis(2-chloroethyl) ether**	10	0.333	♦*Nitroquinoline-1-oxide*	10	0.333
♦**Bis(2-chloroisopropyl) ether**	10	0.333	♦*n-Nitrosodiethylamine*	10	0.333
♦♣**Bis(2-ethylhexyl) phthalate**	10	0.333	♦*n-Nitroso-dimethylamine*	10	0.333
♦**4-Bromophenylphenyl ether**	10	0.333	♦*n-Nitrosodi-n-butylamine*	10	1.67
♦♣**Butyl benzyl phthalate**	10	0.333	♦**n-Nitroso-di-n-propylamine**	10	0.333
Caprolactum	10	0.333	♦**n-Nitroso-diphenylamine and Diphenylamine**	10	0.333
Carbazole	10	0.333	♦*n-Nitrosomethylethylamine*	10	0.333
♦**4-Chloro-3-methylphenol**	10	0.333	♦*n-Nitrosomorpholine*	10	1.67
♦**4-Chloroaniline**	10	0.333	♦*n-Nitrosopiperdine*	10	1.67
♦*Chlorobenzilate*	10	0.333	♦*n-Nitrosopyrrolidine*	10	1.67
1-Chloronaphthalene	10	0.333	*Octadecane*	50	0.333

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**2-Chloronaphthalene**	10	0.333	♦*Parathion*	10	1.67
♦**2-Chlorophenol**	10	0.333	♦*Pentachlorobenzene*	10	1.67
♦**4-Chlorophenylphenyl ether**	10	0.333	♦*Pentachloroethane*	10	0.333
♦♣**Chrysene**	10	0.333	♦*Pentachloronitroben-zene*	10	1.67
♦*cis-Diallate*	10	0.333	♦**Pentachlorophenol**	25	0.833
♦*trans-Diallate*	10	0.333	♦*Phenacetin*	10	1.67
♦**Dibenzofuran**	10	0.333	♦♣**Phenanthrene**	10	0.333
♣*Dibenz(a,h)acridine*	10	0.333	♦♣**Phenol**	10	0.333
Dibenz(a,j)acridine	10	0.333	♦*1,4-Phenylenediamine*	50	0.333
♦♣**Dibenzo(a,h)anthracene**	10	0.333	♦*Phorate*	10	0.333
♦♣**1,2-Dichlorobenzene**	10	0.333	♦*2-Picoline*	10	0.333
♦♣**1,3-Dichlorobenzene**	10	0.333	♦*Pronamide*	10	1.67
♦♣**1,4-Dichlorobenzene**	10	0.333	♦♣**Pyrene**	10	0.333
♦**3,3'-Dichlorobenzidine**	10	0.333	♦♣**Pyridine**	10	0.67
♦**2,4-Dichlorophenol**	10	0.333	♣*Quinoline*	10	0.333
♦*2,6-Dichlorophenol*	20	0.333	♦*Safrole*	10	0.333
3,4-Dichlorophenol	10	0.333	*Terbufos*	50	167
♦♣**Diethyl phthalate**	10	0.333	♦*1,2,4,5-Tetrachloro-benzene*	10	1.67
♦*Dimethoate*	10	1.67	♦*2,3,4,6-Tetrachloro-phenol*	10	0.333
♦*p-Dimethylaminoazobenzene*	10	1.67	♦*Tetraethylpyrophos-phate, Sulfotep*	10	1.67
♦*3,3'-Dimethylbenzidine*	50	0.333	♦*Thionazine*	10	1.67
♦♣*7,12-Dimethylbenz(a)an-thracene*	10	0.333	♣*Thiophenol*	50	1.67
♦*a,a-Dimethylphenethylamine*	50	1.67	♦*o-Toluidine*	10	1.67
♦♣**2,4-Dimethylphenol**	10	0.333	♦**1,2,4-Trichloroben-zene**	10	0.333
♦♣**Dimethyl phthalate**	10	0.333	♦**2,4,5-Trichlorophenol**	10	0..667
♦♣**Di-n-butyl phthalate**	10	0.333	♦**2,4,6-Trichlorophenol**	10	0.333
♦*1,3-Dinitrobenzene*	10	1.67	♦*o,o,o-Triethylphospho-rothioate*	10	1.67
♦**4,6-Dinitro-2-methylphenol**	25	0.833	♦*1,3,5-Trinitrobenzene*	10	0.333
♦♣**2,4-Dinitrophenol**	25	0.833	Acenaphthene, SIM	0.10	0.00333
♦**2,4-Dinitrotoluene**	10	0.333	Acenaphthylene, SIM	0.10	0.00333
♦**2,6-Dinitrotoluene**	10	0.333	Anthracene, SIM	0.10	0.00333
♦♣**Di-n-octyl phthalate**	10	0.333	Benzo(a)anthracene, SIM	0.10	0.00333
♦*Dinoseb*	10	0.333	Benzo(a)pyrene, SIM	0.10	0.00333
1,4-Dioxane	10	0.333	Benzo(b)fluoranthene, SIM	0.10	0.00333
1,2-Diphenylhydrazine	10	0.333	Benzo(g,h,i)perylene, SIM	0.10	0.00333
♦*Disulfoton*	10	1.67	Benzo(k)fluoranthene, SIM	0.10	0.00333
♦*Ethyl methanesulfonate*	10	0.333	Chrysene, SIM	0.10	0.00333
♦*Famphur*	10	0.333	Dibenzo(a,h)anthracene, SIM	0.10	0.00333
♦♣**Fluoranthene**	10	0.333	2,4-Dinitrotoluene, SIM	0.2	0.0067

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**Fluorene**	10	0.333	2,6-Dinitrotoluene, SIM	0.2	0.0067
♦**Hexachlorobenzene**	10	0.333	Fluoranthene, SIM	0.10	0.00333
♦**Hexachlorobutadiene**	10	0.333	Fluorene, SIM	0.10	0.00333
♦**Hexachlorocyclopentadiene**	10	0.333	Indeno(1,2,3-cd)pyrene, SIM	0.10	0.00333
♦**Hexachloroethane**	10	0.333	1-Methylnaphthalene, SIM	0.10	0.00333
♦*Hexachlorophene*	50	3.33	2-Methylnaphthalene, SIM	0.10	0.00333
♦*Hexachloropropene*	50	3.33	Naphthalene, SIM	0.10	0.00333
♦**Indeno(1,2,3-c,d)pyrene**	10	0.333	Phenanthrene, SIM	0.10	0.00333
♣*Indene*	10	1.67	Pyrene, SIM	0.10	0.00333
♦*Isodrin*	10	0.333	California / Missouri DRO	500	20
♦**Isophorone**	10	0.333	Calilfornia / Missouri ORO	500	20

♦ indicates Appendix IX compound ♣Skinner List compound
Bold compounds are reported in a standard list.
Italicized compounds are only available upon special request by this method.
SIM = Selective Ion Monitoring

1.3 The following compounds may require special treatment when being determined by this method:

- Benzidine may be subject to oxidative losses during solvent concentration, and its chromatographic behavior is poor.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- n-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- n-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. Use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation. See SOPs 3510 / NV03-24 for waters, 3550 / NV03-23 and 3541 / NV03-231 for soils and concrete, and 3580 / NV03-106 for oils, and, if necessary, sample cleanup procedures.

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass

spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using at least a multi-point calibration curve.

3.0 Definitions

3.1 Reduced Volume Extraction / Large Volume Injection (RVE/LVI): The option to use a reduced sample volume for extraction combined with a larger volume extract injection on the instrument. Volumes for this option are shown in this document as RVE/LVI in brackets.

3.2 See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This method may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. Be aware of the locations of those zones, and cool them to room temperature prior to working on them.
- The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatography/mass spectrometer/data system
  - Gas chromatograph (HP or Agilent): Analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.
  - Column: 30 m x 0.25 mm ID with a 0.25 µm film thickness silicone-coated fused-silica capillary column (Phenomenex ZB-5, or equivalent) [RVE/LVI: and a 5 m x 0.32 mm ID guard column (Phenomenex 7CG-G000-000-GZ0, or equivalent].
  - Mass spectrometer capable of scanning from 35 to 500 amu every 1 second less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for Decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 2 when 1µL of the GC/MS tuning standard is injected (50 ηg or less of DFTPP).
  - Data system (Chemstation with Enviroquant): A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is also available that allows integrating the abundances in any EICP between specified time or scan-number limits. The EPA/NIST Mass Spectral Library is also available.
  - Suggested operating conditions (may vary by instrument; see maintenance log for current program):

Mass range:	35-500 amu
Scan time:	1 second/scan
Initial temperature:	40°C hold for 2 minutes
Temperature program:	Rate 1: 15°C/minute to 160°C
	Rate 2: 10°C/minute to 320°C
Final temperature:	320°C hold for at least 1.5 minute.
Injector temperature:	240-250°C
Transfer line temperature:	280°C
Source temperature:	According to manufacturer's specifications (nominally 250 – 275°C)
Injector:	Grob-type, split-less
Injection volume:	1 µL [RVE/LVI: 5 µL]
Carrier gas:	Helium at 1 mL/minute

6.2 Supplies

- Microsyringe, 10 µL.
- Balance, analytical, capable of weighing 0.0001 g
- Glass vials, glass with PTFE (polytetrafluoroethylene)-lined screw-caps or crimp tops.
- Volumetric flasks, Class A, appropriate sizes with ground-glass stoppers.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise, indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water, analyte-free.

7.3 Stock Calibration Standards: Commercially prepared, certified stock standards are purchased:

- The primary standard for the typical 8270 compound list is from Ultra Scientific CUS-6150, or equivalent, with the required targets at 200 µg/mL.
- For PAHs by SIM, use Accustandard Z-014G-FL, or equivalent, with the target PAHs at 2000 µg/mL.
- For Appendix IX and miscellaneous compounds, primary source standards are purchased from NSI; equivalent substitutes are acceptable.

Analyte/Analyte Group	NSI Catalog Number	Concentration (µg/mL)
AIX Mix	C-426	2000
Acid Extractables II	C-415	2000
Amines	C-412	2000
Aramite	922-05-02	2000
a,a-Dimethylphenylamine	922-05-02	2000
Benzidines	C-411	2000
BNA II mix	C-413	2000
B/N III mix	C-414	2000
Hexachlorophene	323-03	5000
Sulfonates	C-416	2000
8270 OP Pest	C-417	2000

7.4 Matrix Spike and Laboratory Control Standard contains all targets to be reported on the samples. The same compounds mentioned in Section 7.3 are designated as the SPCCs and CCCs for 8270C.

- For both a long semivolatile list and the PAH list by SIM, purchase as the second source a 100 µg/mL standard, NSI Catalog # c-408-50x, or equivalent.
- For Appendix IX and miscellaneous compounds, these second source standards are acceptable, as well as equivalents:

Analyte/Analyte Group	RestekCatalog Number	Concentration (µg/mL)
AIX #1 Mix	31625	2000
AIX #2 Mix	31806	2000
Calibration Mix	31618	2000
OP Mix	32419	2000

7.5 Internal standard solutions: The internal standards are 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, and Perylene-d_{12}.

- Purchase certified, internal standard at 4000 µg/mL, NSI C-394, or equivalent.

7.6 GC/MS Tuning Standard: A Methylene chloride solution containing 50 µg/mL of Decafluorotriphenylphosphine (DFTPP) is prepared. The standard also contains 50 µg/mL each of 4, 4'-DDT, Pentachlorophenol, and Benzidine to verify injection port inertness and GC column performance.

- Purchase the tuning standard at 1000 µg/mL from Ultra Scientific, Catalog GCM-150, or equivalent.

7.7 Surrogate standards: The surrogates are Phenol-d_5, 2-Fluorophenol, 2,4,6-Tribromophenol, Nitrobenzene-d_5, 2-Fluorobiphenyl, and p-Terphenyl-d_{14}.

- Purchase the acid/base/neutral and PAH SIM surrogates from NSI, C-LIS-7070, or equivalent, at 50 µg/mL.

7.8 Acetone, Hexane, Methylene chloride, Isooctane, Carbon disulfide, Toluene, and other appropriate solvents, commercial source.

7.9 Sodium sulfate for blank and LCS soil matrix.

7.10 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standard solutions must be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Replace after **one year or sooner** if comparison with quality control check samples indicates a problem, or if the vendor specifies an expiration date sooner than one year.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	3 L, amber glass with Teflon®-lined cap	1 L [RWE/LVI: 250 mL]	Cool 0-6°C. Keep in dark.	7 days from collection until extraction, 40 days after extraction	SW-846 Chapter 2
Soil, Oil, Concrete	4 oz. glass jar with Teflon®-lined cap	30 g	Cool 0-6°C.	14 days from collection until extraction, 40 days after extraction	

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

Certain quality control and reporting criteria may vary depending on whether SW-846 8000B or 8000C criteria are required. In these cases, both sets of criteria have been noted in this SOP. 8000C criteria are required to be applied ONLY to Arizona and Washington samples. All other samples must be processed against referenced 8000B criteria. Exceptions may be required on a project-specific basis.

9.1 Sample QC:

The following QC samples are run with each batch of no more than 20 samples.			
QC Check	Frequency	Acceptance Criteria[1]	**Corrective Action[2]**
Method blank	One per analytical prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank.
LCS[6] for all analytes (2nd source)[6]	One[6] per prep batch	See LIMS and footnote 4 below.	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch.[4] If high and target is ND, OK to report.
MS/MSD (2nd source)	One per batch per matrix, if insufficient sample for MS/MSD, qualify data[3]	See LIMS.	None (the LCS is used to evaluate to determine if the batch is acceptable).
Surrogate(s)	Every sample, spike, standard, and blank	See LIMS.[5]	Check system, re-analyze, re-prep[3, 5].

[1]This is a summary of the acceptance criteria.
[2]All abnormalities must be noted on the data, the benchsheet and in LIMS.
[3]If unable to re-prep samples because of insufficient sample volume or the holding time has expired, then place a comment on the benchsheet and in LIMS.
[4]If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prep and re-analyze the batch. If the LCS exceeds the upper control limit AND the samples from that batch is less than the RL, the data is acceptable to report.
[5]If the surrogate % recovery exceeds the upper control limit AND a sample result is positive above the RL, re-prep and re-analyze the batch. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prep the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed.
[6]LCSD is required for AZ, MA, TX, WV.

- A **Method blank** is extracted with every batch of samples.
- A **Laboratory Control Sample** (LCS) is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume (reagent water for water batches, Sodium sulfate for soil batches). It is spiked with the same analytes at the same concentrations as the matrix spike. **All target analytes must meet the LCS QC criteria** (laboratory historical limits in LIMS)**.** However, if the LCS is high, and a target is ND, it is acceptable to report the result.
  - The LCS spike is from a **different source than the calibration standards**. Using the 100 µg/mL LCS/MS/MSD standard:
    - **For Non-SIM batches:**
      - Water: add 500 µL [RVE/LVI: 100 µL] of the standard per liter reagent water before extraction by Method 3510C.
      - Soil: add 500 µL of the standard per 30 gram Sodium sulfate before extraction.
      - TCLP: add 1 mL [RVE/LVI: 200 µL] of the standard/500 mL TCLP extraction fluid before extraction by Method 3510C.
      - The final concentration is 50 µg/mL on column.
    - **For SIM batches:**
      - Water: add 1 mL [RVE/LVI: 200 µL] of a 100 X dilution of the NSI standard per liter reagent water.
      - Soil: add 1 mL of a 100 X dilution of the NSI standard per 30 g Sodium sulfate.
      - The final concentration in the extracts is 1.0 µg/mL.
- **Matrix Spike / Matrix Spike Duplicate**: Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair.
  - The MS/MSD spike is from a **different source** than the calibration standards. Using the 100 µg/mL LCS/MS/MSD standard:
    - **For Non-SIM batches**:

- Water: add 500 µL [RVE/LVI: 100 µL] of the standard per liter client sample.
- Soil: add 500 µL of the standard to 30 g client sample.
- TCLP: add 1 mL [RVE/LVI: 200 µL] of the standard per 500 mL client TCLP extract.
- The final concentration is 50.0 µg/mL on column..

- **For SIM batches**:
  - Water: add 1 mL [RVE/LVI: 200 µL] of a 100 X dilution of the NSI standard per liter reagent water.
  - Soil: add 1 mL of 100 X dilution per 30 g client sample.
  - The final concentration is 1.0 µg/mL on column.

- **Surrogate recoveries:** The laboratory evaluates surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The limits for surrogate recoveries are updated biannually (see TestAmerica Nashville's current Control Limits Manual (CLM)). If any surrogate is outside QC limits, and there is no obvious matrix interference, then re-analyze and/or re-extract the sample. If surrogates are still outside limits, flag the data in LIMS. However, if high and all results are non-detect, results are reportable. If surrogate recoveries are low, re-prep the batch.
  - **For Non-SIM**, add 1000 µL [RVE/LVI: 200 µL] of the surrogate standard at a concentration of 50 µg/mL to each sample and batch QC samples prior to extraction for a 50 µg/mL concentration.
  - **For SIM,** prepare a 1 µg/mL standard (500 µL surrogate standard) to 500 mL in methanol. Add 1.0 mL [RVE/LVI: 200 µL] to samples and QC (blanks, MS/MSD and LCS) prior to extraction. The concentration is 1.0 µg/mL.

9.2 Instrument QC

QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
GC/MS Tuning			
a. Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours.	See below in this section for GC/MS Tuning criteria.	Retune the instrument and verify (instrument maintenance may be needed).
b. Column Breakdown	Prior to initial calibration or Continuing calibration verification, every 12 hours.	Breakdown ratio ≤ 20% (30% for 8270C).	Injector or column maintenance and re-calibration.
c. Tailing Factor	Prior to initial calibration or Continuing calibration verification, every 12 hours.	8270C 8270D Benzidine 3 2 Pentachlorophenol 5 2	Injector or column maintenance and re-calibration.
Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8270C: SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15% If %RSD is > 15%, linear regression $r^2 \geq 0.990$, $r \geq 0.995$. 8270D: The minimum RF for all compounds in Attachment 5 must be met[5]. All targets RSD ≤ 20% or use linear regression.	Correct problem then repeat initial calibration.

QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration.	All analytes within 30% of expected value.	Correct problem then repeat initial calibration.
Initial calibration blank	Immediately after ICV	All analytes < MDL	Correct problem, re-calibrate.
Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	8270C: SPCCs average RF ≥ 0.050 and CCCs: ≤30% difference (when using RFs) or drift (when using least squares regression). Non-CCC < 20% true; up to 4 may be < 40%. 8270D: The minimum RF for all compounds listed in Attachment 4 must be met and the percent difference or drift for each target compound ≤ 20%.	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
Internal Standards	Every sample/standard and blank.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL for CCV. EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. See footnote 4 below.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical manager for advice).
Relative Retention Time Window	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check.
MDL verification (extracted)	Minimum yearly.	Detectible	Re-evaluate MDL standard used and MDL; see the technical manager.

[1]8270 requires DFTPP.
[2]This is a summary of the acceptance criteria.
[3]All abnormalities must be noted on the data, the benchsheet and in LIMS.
[4]Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.
[5]LLCV: If RF is not met at the low-level standard, the criterion for a passing LLCV is detection only and must be run following the CCV.

- **Tuning**

GC/MS Tuning (Full Scan)

- Prior to the analysis of samples or calibration standards, the GC/MS system is hardware-tuned using a 50 ng or less injection of DFTPP (in the GC/MS Tuning Standard).
- The 50 µg/mL standard is prepared by adding 2.8 mL of 1000 µg/mL stock standard to 56 mL Methylene chloride . [RVE/LVI: Use a 5X dilution of this solution.]
- Analyses **must** not begin until the tuning criteria are met, and these criteria must be demonstrated at the beginning of each 12-hour shift. Three options are available for acquiring the spectra for reference to meet the DFTPP tuning requirements:

Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex ±1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does

not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct

Option 2 The entire peak may be averaged and background-corrected. Average scans from 0.1 minute before to 0.1 minute after peak.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is required.

Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass, it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance**.

- All subsequent standards, samples, controls, and blanks associated with a DFTPP tune must use the identical mass spectrometer instrument conditions.
- Use the DFTPP mass intensity criteria as follows as tuning acceptance criteria.

DFTPP Key Ions and Ion Abundance Criteria

Mass	m/z Abundance criteria
51	30-60 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40-60 percent of mass 198.
197	Less than 1 percent of mass 198.
198	Base peak, 100 percent relative abundance.
199	5-9 percent of mass 198.
275	10-30 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than mass 443.
442	Greater than 40 percent of mass 198.
443	17-23 percent of mass 442.

- **Breakdown Standard**: The GC/MS Tuning Standard is also used to assess the injection port inertness by evaluating the degradation of DDT to DDE and DDD. This ratio must **not** exceed 20%; see Section 9.2 for **percent breakdown** calculation. Perform injector or column maintenance and recalibrate if the ratio maximum is exceeded for either compound. The breakdown of DDT is measured **before** verification standards and samples are analyzed and every 12 hours throughout the sequence.
- **Tailing Factor**: To evaluate the GC column, Benzidine and Pentachlorophenol (in the GC/MS Tuning Standard) must be present at their normal responses and evaluated for peak tailing. The Benzidine and Pentachlorophenol tailing factors are calculated by the following equation:

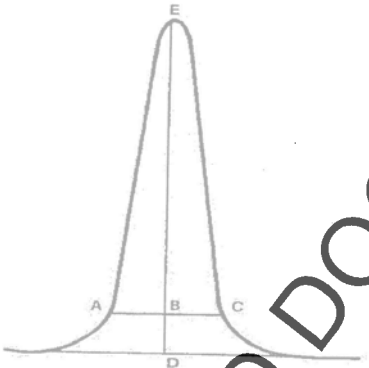
Tailing factor = BC/AB

Maximum Tailing Factor Ratios

Tailing Factor Compounds	8270C	8270D
Benzidine	3	2
Pentachlorophenol	5	2

where the peak is defined as follows: AC is the width at 10% height. DE is the height of peak and B is the height at 10% DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

FIGURE 1
TAILING FACTOR CALCULATION



TAILING FACTOR = $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm
Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

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If all of the specified criteria are met, generate a hardcopy of the spectrum, the mass abundance data and the parameters under which the scans were acquired. This data is filed in the batch for documentation.

GC/MS Tuning (SIM)

- The objective of tuning for conventional full scan analysis is to produce a balanced mass spectrum over the range of interest. The DFTPP tune is, by necessity, done in the full scan mode. However, because the instrument is then immediately switched to the SIM mode, the DFTPP results have limited quality control value. In short, the DFTPP is not analyzed under the same conditions as the calibration, QC, and field samples. In the case of Selective Ion Monitoring (SIM) analysis, there are no comparisons between spectra; instead the instrument is optimized for the relative intensities of the pre-selected analyte ions of interest. For SIM analysis, the laboratory prints out a copy of the autotune (PFTBA) prior to analysis to demonstrate good mass assignment and peak width. No BFB tune is possible while in SIM mode. A printout of the instrument autotune (PFTBA) is included with the data for each day that SIM analyses are run in order to demonstrate good mass assignment and peak width.

- **Calibration**: See Section 10.2.
- **Initial Calibration System Performance Check Compounds (SPCCs)**: A system performance check is performed to ensure that minimum average RFs are met before the calibration curve is used.
  - **For 8270C**: The SPCCs are

System Performance Check Standards (SPCCs)	
Base/Neutral Fraction	**Acid Fraction**
n-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

The **minimum acceptable average RF for the SPCCs is 0.050**. They typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

  - **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
  - If the minimum response factors are not met, the system must be evaluated, and corrective action is taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. **This check must be met before sample analysis begins.** An option is to run a LLCCV to show sensitivity.
- **Initial Calibration Calibration Check Compounds (CCCs) for 8270C only**: The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes. The CCCs are:

Calibration Check Compounds (CCC)	
Base/Neutral Fraction	**Acid Fraction**
Acenaphthene*	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene*	2,4,6-Trichlorophenol
Benzo(a)pyrene*	

*For PAH SIM standard

  - Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte.

Initial Calibration RSD Differences	
8270C	**8270D**
The RSD must be less than or equal to **15%** for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. However, the RSD for each individual CCC must be less than or equal to 30%.	The RSD must be less than or equal to **20%** for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. If not, check

If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.	errors in standard preparation, the possible presence of active sites in the GC system, poor chromatographic behaviors for analytes.

- The **Initial Calibration Verification (ICV)** is a second-source standard run immediately after the initial calibration. The acceptance limits are **70-130%** recovery.
  - Add 250 µL of the second-source standard to 250 µL Methylene chloride in an amber vial to prepare an ICV standard at 50 µg/mL.
  - For PAHs by SIM, use the second-source standard with the target PAHs at 2000 µg/mL. A 10 µg/mL intermediate is made by taking 50 µL of the stock standard along with 20 µL of the base/neutral surrogates. The ICV at 1 µg/mL is made by taking 50 µL of intermediate into 450 µL of Methylene chloride in an amber vial.
  - If ICV acceptance criterion is not met, correct the problem and re-calibrate.
- **Initial Calibration Blank**: a reagent/solvent blank analyzed after the ICV to ensure the system is free of contaminants (< MDL). If not contaminant-free, re-run and/or perform system maintenance.
- The **Continuing Calibration Verification standard (CCV)** is evaluated each day (or every 12 hours) that analysis is performed to determine if the chromatographic system is operating properly.
  - Prepare a daily CCV at 50 µg/mL by adding 100uL of the primary stock solution to 300 µL Methylene chloride in an amber vial. [RVE/LVI: 20 µL to a final volume of 400uL Methylene chloride].
  - For PAHs by SIM, use the primary stock standard with the target PAHs at 2000 µg/mL. A 10 µg/mL intermediate is made by taking 50 µL of the stock standard along with 20 µL of the base/neutral surrogates. A daily CCV at 1 µg/mL is made by taking 50 µL of intermediate into 450 µL of Methylene chloride in an amber vial. [RVE/LVI: 5 µL to a final volume of 500uL of Methylene Chloride].
  - The calibration verification standard is prepared at least weekly and stored at 4°C or less.
  - For 8270C, each **SPCC** in the calibration verification (CCV) standard must meet a **minimum response factor of 0.050**.. **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
  - After the system performance check is met, the **CCCs** are used for 8270C only to check the ongoing validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

CCC % Difference Evaluation Criteria	
8270C	**8270D**
CCCs ≤ 30% and all other target compounds require an RF ≤ 20%; however, up to 5 non-CCC target compounds may be ≤ 40%.	If the percent difference for each target compound is less than or equal to **20%**, then the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift) for any target, then corrective action is taken prior to the analysis of samples. All targets are considered as CCCs.

  - If the CCV criteria cannot be met, a new initial calibration must be generated.
- **Continuing Calibration Blank (CCB):** The CCB is run after each CCV. If the result is not ≤ MDL or ½ RL, correct the problem and re-run.
- **Internal standards** are added to every sample, standard, and QA/QC.

- **Retention time** - The retention times of the internal standards in the continuing calibration verification (CCV) standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than **30 seconds** from that in the mid-point standard level of the **most recent initial calibration** sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- **Response** - If the EICP area for any of the internal standards in the continuing calibration verification (CCV) standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the **most recent initial calibration sequence**, the mass spectrometer must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
  - If the internal standard is within criteria, report the second analysis.
  - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
  - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
- The target analytes are quantitated with specific internal standards as shown in this table:

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
Aniline	Benzoic acid	Acenaphthene
Benzyl alcohol	Bis(2-chloroethoxy) methane	Acenaphthylene
Bis(2-chloroethyl) ether	4-Chloroaniline	2-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
2-Chlorophenol	2,4-Dichlorophenol	Dibenzofuran
1,3-Dichlorobenzene	2,4-Dimethylphenol	Diethyl phthalate
1,4-Dichlorobenzene	Hexachlorobutadiene	Dimethyl phthalate
1,2-Dichlorobenzene	Isophorone	2,4-Dinitrophenol
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene
Hexachloroethane	Naphthalene	2,6-Dinitrotoluene
2-Methylphenol	Nitrobenzene	Fluorene
3,4-Methylphenol	Nitrobenzene-d_8 (surr)	2-Fluorobiphenyl (surr)
n-Nitrosodimethylamine	2-Nitrophenol	Hexachlorocyclopentadiene
n-Nitroso-di-n-propyl- amine	1,2,4-Trichlorobenzene	2-Nitroaniline
Phenol	1-Methylnapthalene	3-Nitroaniline
Phenol-d_5 (surr)	Hexachloropropene	4-Nitroaniline
Pyridine	2,6-Dichlorophenol	4-Nitrophenol
2-Chlorophenol-d_4 (surr)	n-Nitrosodi-n-butylamine	2,4,6-Trichlorophenol
1,2-Dichlorobenzene-d_4 (surr)	1,4-Phenylenediamine	2,4,5-Trichlorophenol
1,4-Dioxane	trans-Isosafrole	1,2-Diphenylhydrazine
Pyridine	1,2,4,5-Tetrachlorobenzene	1,3-Dinitrobenzene
2-Picoline	cis-Isosafrole	Pentachlorobenzene
N-Nitrosomethylethylamine	Safrole	1-Naphthaleneamine
Methyl-methoanesulfonate	1-Chloronaphthalene	2-Naphthaleneamine
n-Nitrosodiethylamine	1,4-Naphthoquinone	2,3,4,6-Tetrachlorophenol
Ethylmethanesulfonate	**Quinoline**	Diphenylamine

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
Pentachloroethane	**Chrysene-d_{12}**	5-Nitro-o-Toluidine
Acetonphenone	**6-Methylchrysene**	trans-Diallate
n-Nitrosopyrrolidine	**Dibenz(a,h)acridine**	cis-Diallate
2-Toluidine	**7,12-Dimethylbenz(a)anthracene**	1,3,5-Trinitrobenzene
n-Nitrosomorpholine		Phenacetin
n-Nitrosopiperidine		4-Aminobiphenyl
2-Butoxyethanol		
Indene		
Thiophenol		
Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
Anthracene	Benzidine	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	Benzo(a)anthracene	Benzo(k)fluoranthene
Di-n-butyl phthalate	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol	Butyl benzyl phthalate	Benzo(a)pyrene
Diphenylamine	Chrysene	Dibenz(a, h)anthracene
Fluoranthene	3,3'-Dichlorobenzidine	Di-n-octyl phthalate
Hexachlorobenzene	Pyrene	Indeno(1,2,3-cd)pyrene
n-Nirosodiphenylamine	Terphenyl-d_{14} (surr)	Dibenz(a,j)acridine
Pentachlorophenol	4,4'Methylenebis(2-chloroaniline)	
Phenanthrene	Aramite	
Carbazole	3-Methylcholanthrene	
Bis (2-ethylhexyl)adipate		
Tribromophenol (surr)		
Thionazin		
Pronamide		
Pentachloronitrobenzene		
Dinoseb		
Sulfotepp		
Phorate		
Dimethoate		
Disulfoton		
4-Nitroquinoline-N-oxide		
Methapyrilene		
Isodrin		
Methyl Parathion		
Benzidine		
Parathion		
Hexachlorophene		
Kepone		
4-Dimethylaminoazobenzene		
Chlorobenzilate		
3,3'-Dimethylbenzidine		
2-Acetylaminofluorene		

(surr)= surrogate

- The internal standards selected permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation. If interferences are noted, use the next most intense ion as the quantitation ion (i. e., for 1, 4-Dichlorobenzene-d_4, use 152 m/z for quantitation).
- Dilute the 4000 µg/mL internal standard by 2x with Methylene chloride. The resulting

solution contains each internal standard mixture at a concentration of 2000 µg/mL. Each 0.5 mL sample extract undergoing analysis is spiked with 10 µL [RVE/LVI: 2µL] of the internal standard solution, resulting in a concentration of 40 µg/mL of each internal standard.

- For SIM, dilute the 2000 µg/mL internal standard mix by 10x with Methylene chloride for a 200 µg/mL standard. Each 0.5 mL of sample extract undergoing analysis is spiked with 10 µL [RVE/LVI: 5 µL] of internal standard solution, resulting in a concentration of 2 µg/mL of each internal standard.

- **Evaluation of target analyte retention time:** The relative retention time (RRT) of each target analyte in each calibration standard must agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement. This criterion is met with the use of a ± 0.25 minute retention time window. Representative retention times are shown in Attachments 1 and 2.
- **Method Detection Limit Verification (MDLV)**: Annually, verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	1000 mL [RVE/LVI: 250 mL]
Soil, Concrete	30 grams
Oil	1 gram

- Samples are nominally prepared by one of the following methods prior to GC/MS analysis:

Matrix	Methods	SOP #
Water	3510	NV03-24
Soil/sediment/Concrete	3541, 3546, 3550	NV03-231, NV03-25
Oily Waste	3580	NV03-106

- QC samples and client samples must be extracted by the same preparation method.
- All calibration standards, QC samples, and client samples are introduced into the GC/MS using the same injection volume, IS and SS concentrations, and instrument conditions.

10.2 Calibration and Daily Continuing Calibration Verification: Refer to SOP Selection of Calibration Points / CA-P-P-002 and Calibration Curves (General) / CA-Q-S-005. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

- Initially and/or daily, evaluate the DFTPP tune criteria (Section 9.2).
- Evaluate the percent breakdown of DDT (Section 9.2).
- Evaluate the tailing factors for Benzidine and Pentachlorophenol (Section 9.2).

Initial calibration

1 Prepare calibration standards at five (minimum) different concentrations.

Concentration (µg/mL)	µL 200 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 200 µg/mL standard/500 µL (5 µL injection)
2	5	1
10	25	5

	20	50	10
	50	125	25
	80	200	40
	100	250	50

- At least one of the calibration standards corresponds to a sample concentration **at or below the laboratory reporting limit (RL)**. The remaining standards correspond to the working range of the GC/MS system.
- Each standard contains each analyte to be reported. These target analytes may not include the entire list of analytes for which the method has been demonstrated; however, the laboratory **must not** report a quantitative result for a target analyte that was not included in the calibration standard(s).
- Surrogates are included at the same concentrations.
- The internal standards are at a constant 40 µg/mL. Each 0.5 mL aliquot of calibration standard is spiked with 10 µL [RVE/LVI: 2 µL] of the internal standard solution prior to analysis.

2 For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:

Concentration (µg/mL)	µL 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 10 µg/mL standard/500 µL(5 µL injection)
0.05*	2.5	0.5
0.1	5	1
0.5	25	5
1	50	10
5	250	50
10	500	100

*The 0.05 µg/mL standard must be used for low-level SIM analysis of samples from Wisconsin.

- Surrogates are included at the same concentrations.
- The internal standards are at a constant 2 µg/mL.
- See Attachments 2 and 3 regarding SIM Mass groups.

3 Analyze 1 µL [RVE/LVI: 5 µL] of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte. See Attachment 1. **Two characteristic ions must be valid for the low standard to be used.**

4 Calculate response factors (RFs) for each target analyte relative to one of the internal standards.

5 Evaluate the **system performance check compounds (SPCCs):** The minimum acceptable average RF for these compounds is 0.050 for 8270C. For 8270D, see Attachment 4. This check must be met before sample analysis begins.

6 Evaluate the **calibration check compounds (CCCs)**: If the RSD of any CCC is greater than 8270C criteria, then correct the chromatographic system reactivity before analysis begins. For 8270D, all compounds are treated as CCCs and must be within ± 20%.

7 Evaluate the **retention times.**

8 Evaluate the **linearity of target analytes** - If the RSD (8270C ± 15%; 8270D ± 20%) of any target analytes is within acceptance limits , then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor is used for quantitation. If the RSD of any target analyte is greater than the acceptance criteria , linear

	regression is used for calibration. The correlation coefficient r^2 must be at least 0.990 ($r \leq 0.995$). If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate. See Section 11 for equations and information on linear regression calibration.
9	Evaluate the intercept; it must be ≤ RL or re-calibrate.
10	Evaluate the success of the initial calibration by running an Initial Calibration Verification (ICV).
11	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.

Initial Calibration Sequence Summary

1	DFTPP Tuning Criteria/DDT Breakdown/Tailing Factors
2	Calibration Standards
3	ICV
4	ICB

Daily continuing calibration verification - Calibration verification is performed at the beginning of **each** 12-hour analytical shift.	
1	The initial calibration for each compound of interest is verified once every 12 hours and prior to sample analysis by analyzing a continuing calibration verification (CCV) standard.
2	Evaluate the **system performance check compounds (SPCCs):** Each SPCC in the calibration verification (CCV) standard must meet the **minimum response factor criteria** for 8270C or 8270D in the initial calibration.
3	Evaluate the **minimum response factors** of each of the most common target analytes in the calibration verification standard (same as SPCCs).
4	Evaluate the **calibration check compounds (CCCs)** for method criteria. For 8270D or for shortened compound lists, all target analytes must meet ± 20% criteria. Use the initial calibration criteria.
5	Evaluate the **internal standard retention times** in the CCV.
6	Evaluate the **internal standard responses**.
7	Analyze an extraction blank after the continuing calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants.

10.3 Sample Analysis: Refer to Acceptable Manual Integration Practices / CA-Q-S-002.

1	Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL [RVE/LVI: 2 µL] of the internal standard solution to the 0.5 mL concentrated sample extract.
2	Inject a 1 µL [RVE/LVI: 5 µL] aliquot of the sample extract into the GC/MS system. The volume to be injected contains 50 ng of base/neutral and 50 ng of acid surrogates (assuming 100% recovery).
3	The recommended sequence for a 20-sample batch is as follows:

1	DFTPP Tuning Criteria /DDT Breakdown/Tailing Factors*
2	CCV
3	Method Blank
4	LCS
5	Matrix Spike
6	Matrix Spike
7	Samples 1-20

*Not used for SIM.

4	If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed in the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 µg/mL, unless a more sensitive GC/MS system is being used, e. g., 2 µg/mL for SIM).
5	Evaluate the specific internal standard response. Dilutions may be required to meet this criterion. Notes: Specific analytes associated with an internal standard within -50 to +100% from the last calibration verification (CCV) may be reported with approval from the supervisor or manager even if other internal standards in that analysis are outside limits. Only analytes associated with the internal standard(s) within limits may be reported from that analysis.
6	The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. Multiple ions are used for compound identification; see Attachment 2. Secondary ions may drop below 30% relative intensity at concentrations less than 1 µg/mL.

10.5 Qualitative analysis

- The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Attachments 1 and 2 list the primary and secondary ions for each analyte. Compounds are identified when the following criteria are met.
- The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. **Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.** When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the ±30% relative intensity, the compound is reported as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair. (See Attachment 1.)
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analyses co-elute (i. e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound. The analyst must

carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. If all of the ions associate with the reference spectrum for the target analyte are present and within the ±30% criteria, a positive result is assumed even in the presence of extraneous ion fragments without presumptive evidence (all ions associated with the target analyte are also present in the interfering peak) for a negative identification.

- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights for 8270C and 50% of the average of the two peak heights for 8270D samples. Mathematically, the two equations used are equivalent. Verification is performed on a mid-level control each day of use. Otherwise, structural isomers are identified as isomeric pairs. (See Attachment 1.)
- For samples containing components not associated with the calibration standards or the requested target list, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
  - For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:
    1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) are present in the sample spectrum.
    2) The relative intensities of the major ions agree within ±20%. **Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.**
    3) Molecular ions present in the reference spectrum are present in the sample spectrum.
    4) Ions present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of co-eluting compounds.
    5) Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance of the primary characteristic ion from the EICP.
- If the RSD of a compound's response factor is 15% for 8270C and 20% for 8270D, or less, then the concentration in the extract is determined using the average response factor (RF) from initial calibration data. If greater than the criteria , use linear regression.
- Where applicable, the concentration of any non-target compounds identified in the sample is estimated. The same formulae are used with the following modifications: The areas A_x and A_t are from the total ion chromatograms, and the RF for the compound is assumed to be 1.
- The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.
-

10.7 Instrument Maintenance

Careful examination of the standard chromatogram indicates whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. Re-calibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. In addition, significant maintenance activities or hardware changes may also require re-calibration. These significant maintenance activities include, changing, replacing, or reversing the column; cleaning the MS source; changing the electron multiplier; or injector port.

11.0 Calculations / Data Reduction

11.1 Accuracy

% Recovery = Measured concentration x 100
Known concentration

11.2 Precision (RPD)

RPD = Absolute value (orig. sample value - dup. sample value) x 100
(Orig. sample value + dup. sample value)/2

11.3 Breakdown Calculation:

% Breakdown of DDT = Sum of degradation peak areas (DDD + DDE) x 100
Sum of all peak areas (DDT + DDE + DDD)

1.4 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

11.5 Mean Response Factor, Standard Deviation, Relative Standard Deviation

$$RF_{mean} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \frac{\sum_{i=1}^{n} \left(RF_i - RF_{mean}\right)^2}{n-1}$$

$$RSD = \frac{SD \times 100}{RF_{mean}}$$

11.6 % Difference, % Drift

$$\% \text{ Difference} = \frac{(RF_v) - (\text{Avg. RF}) \times 100}{(\text{Avg. RF})}$$

RF_v = RF from verification standard
Avg. RF = Average RF from Initial Calibration.

$$\% \text{ Drift} = \frac{\text{Result - True Value} \times 100}{\text{True Value}}$$

11.7 Linear Calibration Using a Least Squares Regression: This approach is not used for analytes that meet the RSD limits. For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

by minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y_i', the response calculated from the constructed equation). The regression equation is:

$y_i' = ax_i + b$

a = regression coefficient or the slope of the line.
b = the y-intercept.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
x_I = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(y_i - y_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i (y_i - y_i')^2$$

w_i = weighting factor for the i^{th} calibration standard (w=1 for unweighted least squares regression).

y_i – observed instrument response (area) for the i^{th} calibration standard.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used. Examples of allowed weighting factors which can place more emphasis on numbers of smaller value are:

$w_i - 1/x_i$ or $w_i = 1/x_i^2$

Do not include the origin (0, 0) as an extra calibration point. Reprocess each calibration standard as an unknown to determine the best fit model. Each calibration point above the RL must be ± 15% true (8000B) or ±20% true (8000C); the RL-level standard must be ± 30% true.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.990$.

11.8 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response or Response ratio
x = Concentration

11.9 Calculation

- For **aqueous** samples:

$$\textbf{Concentration (µg/L)} = \frac{A_x V_t D}{RF_{mean} V_s} \quad \text{or} \quad \frac{(\mu g/mL \text{ from instrument})(D)(1000)}{mL \text{ extracted}}$$

A_x = Area of the peak for the analyte in the sample.
V_t = Total volume of the concentrated extract (mL).
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
RF_{mean} = Mean response factor from the initial calibration (area/concentration).
V_s = Volume of the aqueous sample extracted in mL.

- For **non-aqueous** samples:

$$\textbf{Concentration (µg/kg)} = \frac{A_x V_t D}{RF_{mean} W_s} \quad \text{or} \quad \frac{(\mu g/mL \text{ from instrument})(D)(1000)}{g \text{ extracted}}$$

A_x, V_t, D, RF_{mean} are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Dispose of waste extracts in the waste solvent drum.

15.0 References / Cross References

15.1 Method 8270C, SW-846 Update III Revision 3, December 1996 and **Method 8270D**, Update IV, Revision 4, February 2007.

15.2 Method 8000B, SW-846, Revision 2, December 1996, **Method 8000C**, Revision 3, March 2003.

15.3 TestAmerica Nashville's Quality Assurance Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002, Calibration Curves (General) / CA-Q-S-005, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method

Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, 3550 / NV03-23, and 3510 / NV03-24, 3541 / NV03-231, 3580 / NV03-106,8270/NVOH04-22.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

15.7 Corporate Quality Memorandum No. CA-Q-QM-005, May 19, 2010.

16.0 Method Modifications

Item	Modification
1	See Attachment 5 for the State of Ohio specific criteria.
2	See Attachment 6 for the State of Missouri DRO, CA LUFT DRO by GC/MS.
3	Verify with state certifications the correct version of this method to report. **Analyze and report by 8270D for Canadian, NJ, NC, OK, SC, and WV samples.**
4	SIM is not allowed for South Carolina samples unless pre-approved by the state on a project-specific basis.

17.0 Attachments

Attachment 1, Characteristic Ions for Semivolatile Compounds[a]

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
1,4-Dioxane	2.568	88	58
n-Nitrosodimethylamine	2.700	74	42, 44
Pyridine	2.714	79	52
2-Picoline	3.464	93	66, 92
n-Nitrosomethylethylamine	3.558	88	42, 43, 56
2-Fluorophenol (surr)	3.687	112	64
Methyl methanesulfonate	3.764	80	79, 65, 95
n-Nitrosodiethylamine	4.009	102	42, 57, 44, 56
Ethyl methanesulfonate	4.197	79	109, 97, 45, 65
Hexachloropropene	4.261	213	211,215,117,106,141
Phenol-d_5 (surr)	4.266	99	42, 71
Aniline	4.270	93	66, 65
Bis(2-chloroethyl) ether	4.294	93	63, 95
Phenol	4.275	94	65, 66
2-Chlorophenol	4.345	128	64, 130
1,3-Dichlorobenzene	4.425	146	148, 113
1,4-Dichlorobenzene-d_4 (IS)	4.444	152	150, 115
1,4-Dichlorobenzene	4.454	146	148, 113
Pentachloroethane	4.474	117	165, 167, 119
Benzyl alcohol	4.543	79	108, 77
n-Decane	4.550	57	
1,2-Dichlorobenzene	4.571	146	148, 113
2-Methylphenol	4.628	108	107, 77, 79, 90
Bis(2-chloroisopropyl) ether	4.632	45	77, 79
N-Nitrosodi-n-propylamine	4.717	130	42, 101, 70
3, 4-Methylphenol	4.717	107	108, 77, 79, 90
Hexachloroethane	4.764	117	201, 199
Nitrobenzene-d_5 (surr)	4.806	82	128, 54
Nitrobenzene	4.816	77	123, 65
n-Nitrosopyrrolidine	4.907	102	41, 42, 68, 69
Acetophenone	4.912	105	71, 51, 120
n-Nitrosomorpholine	4.916	108	116, 86
o-Toluidine	4.940	106	107, 77, 51, 79

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Isophorone	4.957	82	95, 138
2-Nitrophenol	5.018	139	109, 65
2,4-Dimethylphenol	5.037	122	107, 121
Bis(2-chloroethoxy)methane	5.088	93	95, 123
n-Nitrosopiperidine	5.114	114	42, 55, 56, 41
Benzoic acid	5.116	105	122, 77
2,4-Dichlorophenol	5.168	162	164, 98
1,2,4-Trichlorobenzene	5.215	180	182, 145
Naphthalene-d_8 (IS)	5.248	136	68
Naphthalene	5.257	128	129, 127
o,o,o-Triethylphosphorthioate	5.302	198	80, 53, 54164, 63
4-Chloroaniline	5.304	127	129, 65, 92
Hexachlorobutadiene	5.370	225	223, 227
a,a-Dimethylphenethylamine	5.372	58	91, 65, 134, 42
2,6-Dichlorophenol	5.523	162	
Hexachloropropene	5.556	213	211, 215, 117, 106, 141
4-Chloro-3-methylphenol	5.615	142	107, 144
2-Methylnaphthalene	5.704	142	141
n-Nitrosodi-n-butylamine	5.729	84	57, 41, 116, 158
1,4-Phenylenediamine	5.734	198	80, 53, 54, 52
1-Methylnaphthalene	5.779	142	141, 115
Hexachlorocyclopentadiene	5.854	237	235, 272
Isosafrole (trans)	5.861	162	131, 104, 77
2,4,6-Trichlorophenol	5.911	196	198, 200
2,4,5-Trichlorophenol	5.914	196	198, 97, 132, 99
2-Fluorobiphenyl (surr)	5.953	172	171
2-Chloronaphthalene	6.003	162	127, 164
Isosafrole (cis)	6.054	162	131, 104, 77
1,2,4,5-Tetrachlorobenzene	6.063	216	214,179,108,143,218
2-Nitroaniline	6.118	138	92, 65
2,3-Dichloroaniline	6.134	161	90, 63
Safrole	6.204	162	104, 77, 103, 135
Dimethyl phthalate	6.245	163	194, 164
1-Chloronaphthalene	6.284	162	127, 164
2,6-Dinitrotoluene	6.296	165	63,89, 121
Acenaphthylene	6.320	152	151, 153
1,4-Naphthoquinone	6.374	158	104, 102, 76, 50, 130
3-Nitroaniline	6.404	138	108, 92
Acenaphthene	6.447	154	153, 152
2,4-Dinitrophenol	6.470	184	63, 154
1,3-Dinitrobenzene	6.486	168	76, 50, 75, 92, 122
4-Nitrophenol	6.527	65	109, 139
Dibenzofuran	6.560	168	139
2,4-Dinitrotoluene	6.574	165	63, 89, 182
Acenaphthene-d_{10} (IS)	6.656	164	162, 160
Diethyl phthalate	6.738	149	177, 150
4-Chlorophenyl phenyl ether	6.790	204	206, 141
Fluorene	6.799	166	165,167
Pentachlorobenzene	6.806	250	252,108,248,215,254
4-Nitroaniline	6.837	138	65, 108, 92, 80, 39
1-Naphthylamine	6.844	143	115, 89, 63

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
4,6-Dinitro-2-methylphenol	6.865	198	51, 105, 182, 77
n-Nitrosodiphenylamine	6.879	169	168, 167
2-Naphthylamine	6.895	143	115, 116
2,3,4,6-Tetrachlorophenol	6.900	232	131,230,166,234,168
1,2-Diphenylhydrazine	6.903	77	105, 182
2,4,6-Tribromophenol (surr)	6.987	330	332, 141
Thionazine	7.027	107	96, 97,143, 79, 68
5-Nitro-o-toluidine	7.051	152	77, 79, 106, 94
Diphenylamine	7.107	168	169, 167
4-Bromophenyl phenyl ether	7.138	248	250, 141
Hexachlorobenzene	7.255	284	142, 249
Sulfotepp	7.276	322	97, 202
1,3,5-Trinitrobenzene	7.314	213	74, 120, 91, 63
Diallate (trans)	7.337	86	234, 43, 70
Phenacetin	7.337	108	180,179,109,137,80
Phorate	7.347	75	121, 97, 93, 260
Pentachlorophenol	7.387	266	264, 268
Diallate (cis)	7.403	86	234, 43, 70
Dimethoate	7.474	87	93, 125, 143, 229
Phenanthrene-d_{10} (IS)	7.476	188	94, 80
Phenanthrene	7.495	178	179, 176
Anthracene	7.528	178	176, 179
4-Aminobiphenyl	7.568	169	168, 170, 115
n-Octadecane	7.586	58	71, 85
Pronamide	7.619	173	175, 145, 109, 147
Carbazole	7.641	167	139, 84
Pentachloronitrobenzene	7.676	237	142,214,249,295,265
Disulfoton	7.723	88	97, 89, 142, 186
Dinoseb	7.737	211	163, 147, 117, 240
Di-n-butyl phthalate	7.914	149	150, 104
Methyl parathion	8.000	109	125, 263, 79, 93
Parathion	8.292	109	97, 291, 139, 155
4-Nitroquinoline-1-oxide	8.310	190	101, 128, 75, 116
Methapyrilene	8.371	58	50, 191, 71
Fluoranthene	8.374	202	100, 101, 203
Benzidine	8.464	184	92, 185
Isodrin	8.522	193	66, 195, 263,265,147
Pyrene	8.543	202	100, 101, 200, 203
Terphenyl-d_4 (surr)	8.652	244	122, 212
Aramite	8.870	191	319, 334, 197, 321
Dimethylaminoazobenzene	9.001	120	77, 105, 148, 42
Butyl benzyl phthalate	9.028	149	91, 206
Chlorobenzilate	9.034	139	253, 111, 141
Hexachlorophene	9.070	185	209,406
3,3'-Dimethylbenzidine	9.251	212	106, 196, 180
Bis (2-ethylhexyl) adipate	9.298	129	57, 112, 147
4,4'-Methylenebis (2-chloroaniline)	9.301	231	266, 140, 77
Kepone	9.316	272	274,237,178,143,270
3,3'-Dichlorobenzidine	9.423	252	254, 126
Benz(a)anthracene	9.446	228	229, 226
2-Acetylaminofluorene	9.453	181	180, 223, 152

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Chrysene-d_{12} (IS)	9.456	240	120, 36
Chrysene	9.474	228	226, 229
Bis(2-ethylhexyl) phthalate	9.474	149	167, 279
Di-n-octyl phthalate	9.926	149	167, 43
Benzo(b)fluoranthene	10.292	252	253, 125
3-Methylcholanthrene	11.305	268	252,253,126,134,113
Benzo(k)fluoranthene	10.311	252	253, 125
Benzo(a)pyrene	10.579	252	253, 125
7,12-Dimethylbenz(a)anthracene	10.600	256	241, 239, 120
Perylene-d_{12} (IS)	10.631	264	260, 265
Indeno)1,2,3-c,d)pyrene	11.778	276	138, 277
Dibenz(a,h)anthracene	11.783	278	139, 279
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Benzo(g,h,i)perylene	12.107	276	138, 277
IS = internal standard			
surr = surrogate			
[a]See Attachment 2 for Retention Times and Ions used with SIM			

Attachment 2, Characteristic Ions for PAH Compounds Using SIM

Compounds	RT	Primary	Secondary*
1,4-Dichlorobenzene-d_4	6.66	152	
2-Fluorophenol	5.471	112	64
Phenol-d_5	6.251	99	71.1
Naphthalene-d_8	8.24	136	
Nitrobenzene-d_5	7.32	82.1	128.1
Naphthalene	8.27	128.1	129.1
2-Methylnaphthalene	9.13	142.1	141.1
1-Methylnaphthalene	9.42	142.1	141.1
Acenaphthene-d_{10}	10.91	164.1	
2-Fluorobiphenyl	9.82	172.1	
Acenaphthylene	10.67	153	151.1
Acenaphthene	10.958	15.1	154.1
Fluorene	11.7	166.1	167.1
Phenanthrene-d_{10}	13.3	188	
2,4,6-Tribromophenol	12.167	329.8	331.8
Phenanthrene	13.33	178.2	176.2
Anthracene	13.4	178.2	176.2
Fluoranthene	15.28	202.2	101.1
Chrysene-d_{12}	17.64	240.1	
Pyrene	15.66	202.2	101.1
Terphenyl-d_{14}	15.89	244.2	
Benzo(a)anthracene	17.61	228.2	229.2
Chrysene	17.68	228.2	229.2
Perylene-d_{12}	20.2	264.2	
Benzo(b)fluoranthene	19.45	252.2	126.1
Benzo(k)fluoranthene	19.49	252.2	126.1
Benzo(a)pyrene	20.08	252.2	126.1
Indeno(1,2,3-cd)pyrene	22.69	276.2	277.2
Dibenzo(ah)anthracene	22.7	278.2	279.2

Compounds	RT	Primary	Secondary*
Benzo(ghi)perylene	23.43	276.2	277.2
Internal standards are in **bold.**			

Attachment 3, SIM Mass Groups

Mass Group	Compound	RT	Primary	Secondary	Dwell Time
1	2-Fluorophenol	5.42	112	64	25 ms
	Phenol-d_5	6.2	99	71	
	1,4-Dichlorobenzene-d_4	6.6	152		
	Nitrobenzene-d_5	7.26	82.1	128.1	
4.20 min.					
2	Naphthalene-d_8	8.17	136		50 ms
	Naphthalene	8.2	128.1	129.1	
	2-Methylnaphthalene	9.19	142	141.1	
	1-Methylnaphthalene	9.35	142.1	141.1	
	2-Fluorobyphenyl	9.75	172.1		
5.35 min.					
3	Acenaphthylene	10.6	152.1	151.1	25 ms
	Acenaphthalene-d_{10}	10.83	164.1		
	Acenaphthene	10.88	153.1	154.1	
	Fluorene	11.69	166.1	167.1	
	2,4,6-Tribromophenol	12.11	329.8	331.8	
6.55 min.					
4	Phenanthrene-d_{10}	13.21	188		50 ms
	Phenanthrene	13.25	178.2	176.2	
	Anthracene	13.32	178.2	176.2	
	Fluoranthene	15.21	202.2	101.1	
	Pyrene	15.58	202.2	101.1	
	Terphenyl-d_{14}	15.81	244.2		
7.75 min.					
5	Benzo(a)anthracene	17.52	228.2	229.2	100 ms
	Chrysene-d_{12}	17.55	240.1		
	Chrysene	17.59	228.2	229.2	
9.85 min.					
6	Benzo(b)fluoranthene	19.34	252.2	126.1	100 ms
	Benzo(k)fluoranthene	19.38	252.2	126.1	
	Benzo(a)pyrene	19.96	252.2	126.1	
	Perylene-d_{12}	20.07	264.2		
10.65 min.					
7	Indeno(1,2,3-cd)pyrene	22.5	276.2	277.2	100 ms
	Dibenzo(a,h)anthracene	22.52	278.2	279.2	
	Benzo(g,h,i)perylene	23.21	276.2	277.2	
12.20 min.					

Attachment 4, 8270D Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Attachments 1 and 2.

Compound	Minimum RF	Compound	Minimum RF
Benzaldehyde	0.010	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800

Compound	Minimum RF	Compound	Minimum RF
Bis(2-chloroethyl)ether	0.700	2,4-Dinitrotoluene	0.200
2-Chlorophenol	0.800	Diethyl phthalate	0.010
2-Methylphenol	0.700	1,2,4,5-tetrachlorobenzene	0.010
2,2'-Oxybis-(1-chloropropane)	0.010	4-Chlorophenyl-phenyl ether	0.400
Acetophenone	0.010	Fluorene	0.900
4-Methylphenol	0.600	4-Nitroaniline	0.010
n-Nitroso-di-n-propylamine	0.500	4,6-Dinitro-2-methylphenol	0.010
Hexachloroethane	0.300	4-Bromophenyl-phenyl ether	0.100
Nitrobenzene	0.200	n-Nitrosodiphenylamine	0.010
Isophorone	0.400	Hexachlorobenzene	0.100
2-Nitrophenol	0.100	Atrazine	0.010
2, 4-Dimethylphenol	0.200	Pentachlorophenol	0.050
Bis(2-chloroethoxy)methane	0.300	Phenanthrene	0.700
2,4-Dichlorophenol	0.200	Anthracene	0.700
Naphthalene	0.700	Carbazole	0.010
4-Chloroaniline	0.010	Di-n-butyl phthalate	0.010
Hexachlorobutadiene	0.010	Fluoranthene	0.600
Caprolactam	0.010	Pyrene	0.600
4-Chloro-3-methylphenol	0.200	Butyl benzyl phthalate	0.010
2-Methylnaphthalene	0.400	3,3'-Dichlorobenzidine	0.010
Hexachlorocyclopentadiene	0.050	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis-(2-ethylhexyl)phthalate	0.010
1,1'-Biphenyl	0.010	Di-n-octyl phthalate	0.010
2-Chloronaphthalene	0.800	Benzo(b)fluoranthene	0.700
2-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(a)pyrene	0.700
2,6-Dinitrotoluene	0.200	Indeno(1,2,3-cd)pyrene	0.500
Acenaphthylene	0.900	Dibenz(a,h)anthracene	0.400
3-Nitroaniline	0.010	Benzo(g,h,i)perylene	0.500
Acenaphthene	0.900	2,3,4,6-Tetrachlorophenol	0.010
2,4-Dinitrophenol	0.010		

Attachment 5, State of Ohio Specific Criteria.
Only those compounds in the original EPA Method 8270C may be reported. Any compounds in this SOP in italics in Section 1 are not part of the original 8270C method. Run Ohio VAP samples according to SOP 8270/NVOH04-22.

Attachment 6, Missouri Department of Natural Resources (and CA LUFT) require(s) that **DRO** be analyzed by GC/MS.

- Tuning and frequency requirements are the same as in 8270, omitting DDT, Pentachlorophenol, and Benzidine.
- Extract water samples per SOP 3510 / SA03-24 and solid samples per SOP 3550 / SA03-23.
- Only base/neutral surrogates are needed.
- GC/MS mass range is 35-550 ηmu.
- Use a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 1,000 µg/mL each in Methylene chloride.

- Retention time windows set using C_{10}, C_{21}, and C_{35}. For DRO, set RT 0.1 minutes after C10 to 0.1 minutes after C21. For ORO, set RT 0.1 minutes after C_{21} to 0.1 minutes after C_{35}. Verify RT daily (24 hours) by running component standard.
- Quantitative using baseline-to-baseline, not valley-to-valley. The Total Ion Chromatogram must be used to quantitate.
- The Response Factor determined for DRO (C_{10}-C_{21}) must be used for C_{21}-C_{35}.
- Subtract area from any Internal Standard and surrogates.
- % RSD ≤ 20.
- Run a CCV at the beginning and end of each batch; it must contain all targets reported, at mid-point of calibration, % D ≤ 20.
- Run a Method Blank every extraction batch, and LCS and MS/MSD per batch.
- May reprocess file to quantitate PAH if needed. For individual targets, % RSD ≤ 15.
- Quantitation of DRO must be by external standard.

18.0 Revision History

- Revision 12, 22 October 2008
  - Integration for TestAmerica and STL operations.
  - Insert corrective action procedures
  - To incorporate Update IV criteria.
- Revision 13, 9 October 2009
  - Consolidation of text, general editing.
  - Add Appendix IX and miscellaneous compound details.
  - Distinguish 8270C versus 8270D.
- Revision 14, 30 September 2011
  - Organizational changes.
  - Add amendments 13a and 13b.
  - Add reference to SOP 3541 for concrete and SOP Calibration Curves (General).
  - Add QAF-45 and Section 14.2.
  - Remove WY as a state requiring QC every 10 samples.
  - Change Attachment 5 to refer analysts to OH8270 SOP.
  - Add Attachment 7
  - Add option to run LLCCV to show sensitivity.
  - Add note about low-level calibration standard for SIM WI samples.
  - Lower several report limits.
  - Specify GC resolution between two isomer peaks for 8270C versus 8270D.
- Revision 15, 31 December 2012
  - Organizational changes.
  - Incorporation of amendments 14a, b, c.
  - OK no longer limits batch size to 10 samples.
  - Specify that $r^2 \geq 0.990$.
  - Substitute LIMS for the Control Limits Manual.
  - Distinguish between the RSD maximum for 8270C and 8270D. For 8270D, all targets are treated as CCCs.
  - Add re-fitting text to the linear calibration section.
  - Add Reduced Volume Extraction / Large Volume Injection (RVE / LVI).

Title: VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) SW-846 METHOD 8260B/C

Approvals (Signature/Date)

John Haley	8/7/13	Glenn L. Norton	8/5/13
John Haley Department Supervisor	Date	Glenn Norton Volatiles Operations Manager	Date
Michael H. Dunn	8/30/13	Johnny Davis	8/15/13
Michael H. Dunn\ Technical Director Quality Assurance Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date

1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine volatile organic compounds in a variety of matrices; it is applicable to nearly all types of samples, regardless of water content, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

CAS No. (a)	Compound	CAS No. (a)	Compound
630-20-6	1,1,1,2-Tetrachloroethane[1, 2, 6, 7]	156-59-4	cis-1,2-Dichloroethene[1, 2, 5, 6, 7]
71-55-6	1,1,1-Trichloroethane[1, 2, 5, 6]	10061-01-5	cis-1,3-Dichloropropene[1, 2, 5, 6, 7]
79-34-5	1,1,2,2-Tetrachloroethane[1, 2, 5, 6, 7]	110-82-7	Cyclohexane[4, 5]
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane[5,6,7]	108-94-1	Cyclohexanone[4]
79-00-5	1,1,2-Trichloroethane[1, 2, 5, 6]	74-95-3	Dibromomethane[1, 2, 6, 7]
75-34-3	1,1-Dichloroethane[1, 2, 5, 6, 7]	75-27-4	Dichlorobromomethane[1,2, 6, 7]
75-35-4	1,1-Dichloroethene[1, 2, 5, 6, 7]	75-71-8	Dichlorodifluoromethane[1, 2, 5, 7]
563-58-6	1,1-Dichloropropene[1, 7]	75-43-4	Dichlorofluoromethane[4]
87-61-6	1,2,3-Trichlorobenzene[1]	64-17-5	Ethanol[3]
96-18-4	1,2,3-Trichloropropane[1, 2, 6, 7]	141-78-6	Ethyl acetate [4]
526-73-8	1,2,3-Trimethylbenzene	140-88-5	Ethyl acrylate
120-82-1	1,2,4-Trichlorobenzene[1, 2, 5]	60-29-7	Ethyl ether (Diethyl ether)[4]
95-63-6	1,2,4-Trimethylbenzene[1, 9]	97-63-2	Ethyl methacrylate[2, 7]
96-12-8	1,2-Dibromo-3-chloropropane[1, 2, 5, 7]	100-41-4	Ethylbenzene[1, 2, 5, 6, 7, 8, 9]
95-50-1	1,2-Dichlorobenzene[1, 2, 5, 6, 7]	106-93-4	Ethylene dibromide (EDB, 1,2-Dibromoethane)[2]
107-06-2	1,2-Dichloroethane[1, 2, 5, 6, 7, 8]	87-68-3	Hexachlorobutadiene[1, 2]
78-87-5	1,2-Dichloropropane[1, 2, 5, 6, 7]	110-54-3	Hexane[4]
176-02-8	1,3,5-Trichlorobenzene[4]	74-88-4	Iodomethane[2, 6, 7]
108-67-8	1,3,5-Trimethylbenzene[1, 9]	78-83-1	Isobutyl alcohol [2, 7]
541-73-1	1,3-Dichlorobenzene[1, 2, 5, 7]	67-63-0	Isopropy alcohol[4]
142-28-9	1,3-Dichloropropane[1, 7]	180-20-3	Isopropyl ether (IPE, Di-isopropyl ether)[3]
106-46-7	1,4-Dichlorobenzene[1, 2, 5, 6, 7]	98-82-8	Isopropylbenzene (Cumene)[1, 5, 9]
123-91-1	1,4-Dioxane[2, 8]	126-98-7	Methacrylonitrile[2, 7]
590-20-7	2,2-Dichloropropane[1, 7]	79-20-9	Methyl acetate[5]
78-93-3	2-Butanone (MEK)[1, 2, 5, 6, 7, 8]	80-62-6	Methyl methacrylate[2, 7]
126-99-8	2-Chloro-1,3-butadiene (Chloroprene)[2, 7]	1634-04-4	Methyl-t-butyl ether[1, 3, 4, 5, 9]
110-75-8	2-Chloroethyl vinylether[4]	108-87-2	Methylcyclohexane[5]
95-49-8	2-Chlorotoluene[1]	75-09-2	Methylene chloride[1, 2, 5, 6, 7]
591-78-6	2-Hexanone[1, 2, 5, 6, 7]	108-38-3	m-Xylene [9]
75-65-0	2-Methyl-2-propanol (tert-Butyl Alcohol)[3]	91-20-3	Naphthalene[1, 2, 9]

CAS No. (a)	Compound	CAS No. (a)	Compound
91-57-6	2-Methylnapthalene[4]	71-36-3	n-Butanol (n-Butyl Alcohol)[4]
79-46-9	2-Nitropropane[4]	123-86-4	n-Butyl acetate[4]
624-95-3	3,3-Dimethyl-1-butanol[3]	104-51-8	n-Butylbenzene[1, 9]
107-05-1	3-Chloro-1-propene (Allyl chloride)[2,7]	142-82-5	n-Heptane[4]
106-43-4	4-Chlorotoluene[1]	103-65-1	n-Propylbenzene[1, 9]
99-87-6	4-Isopropyltoluene (p-Isopropyltoluene)[1, 9]	95-47-6	o-Xylene[9]
108-10-1	4-Methyl-2-pentanone (MIBK)[1, 2, 5, 6, 7]	76-01-7	Pentachloroethane
67-64-1	Acetone[1, 2, 5, 6, 7]	107-12-0	Propionitrile[2, 7]
75-05-8	Acetonitrile[2, 7]	135-98-8	sec-Butylbenzene[1, 9]
107-02-8	Acrolein (Propenal)[2, 7]	100-42-5	Styrene[1, 2, 5, 6, 7]
107-13-1	Acrylonitrile[2, 6, 7]	75-85-4	tert-Amyl-alcohol (TAA)[3]
71-43-2	Benzene[1, 2, 5, 6, 7, 8, 9]	994-05-8	tert-Amyl-methyl-ether (TAME)[3]
100-44-7	Benzyl chloride[4]	637-92-3	tert-Butyl ethyl ether (Ethyl-tert-butyl-ether, ETBE)[3]
108-86-1	Bromobenzene[1]	762-75-4	tert-Butyl-formate (TBF)[3]
75-25-2	Bromoform[1, 2, 5, 6, 7]	98-06-6	tert-Butylbenzene[1, 9]
74-83-9	Bromomethane[1, 2, 5, 6, 7]	127-18-4	Tetrachloroethene[1, 2, 5, 6, 7]
106-99-0	Butadiene	109-99-9	Tetrahydrofuran[4]
STL00350	C4-C12	108-88-3	Toluene[1, 2, 5, 6, 7, 8, 9]
80006-61-9	C6-C10	156-60-5	trans-1,2-Dichloroethene[1, 2, 5, 6, 7]
75-15-0	Carbon disulfide[1, 2, 5, 6, 7, 8]	10061-02-6	trans-1,3-Dichloropropene[1, 2, 5, 6, 7]
56-23-5	Carbon tetrachloride[1, 2, 5, 6, 7]	110-57-6	trans-1,4-Dichloro-2-butene[2, 6, 7]
108-90-7	Chlorobenzene[1, 2, 5, 6, 7, 8]	79-01-6	Trichloroethene[1, 2, 5, 6]
74-97-5	Chlorobromomethane[1, 6, 7]	75-69-4	Trichlorofluoromethane[1, 2, 5, 6, 7]
124-48-1	Chlorodibromomethane[1, 2, 5, 6, 7]	108-05-4	Vinyl acetate[2, 6, 7]
75-00-3	Chloroethane[1, 2, 5, 6, 7]	75-01-4	Vinyl chloride[1, 2, 5, 6, 7]
67-66-3	Chloroform[1, 2, 5, 6, 7, 8]	1330-20-7	Xylene (total)[1, 2, .5, 6, 7, 8, 9]
74-87-3	Chloromethane[1, 2, 5, 6, 7]		

[1] - Laboratory normal 8260 compound
[2] - Appendix IX compound
[3] - Oxygenate
[4] - Additional compounds by request only
[5] TCL list (OLM 04.2 list)
[6] - Appendix I compound
[7] -Appendix II compound
[8] - Skinner list
[9] - NY Stars List
a = Chemical Abstract Service Registry Number0

1.2 Reporting Limits: The RL for an individual compound is instrument-dependent and also dependent on the choice of sample preparation/introduction method. The RL analyte concentration is defined by the lowest non-zero standard in the calibration curve. Using standard quadrapole instrumentation and the purge-and-trap technique, RLs, though highly matrix-dependent, are provided in the table below for guidance and may not be achievable. RLs listed

for soil are based on wet weight. When reported on a dry weight basis, RLs are higher, based on the percent dry weight in each sample. For the most current RL, refer to LIMS.

Chromatographic Retention Times and Typical Reporting Limits

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
Dichlorodifluoromethane	1.884	1	0.5	2
Chloromethane	2.095	1	0.5	2
Vinyl chloride	2.222	1	0.5	2
Butadiene	2.264	1	0.5	2
Bromomethane	2.601	1	0.5	2
tert-Butyl-formate (TBF)[3]	2.609	20	20	20
Chloroethane	2.718	1	0.5	2
tert-Amyl-alcohol (TAA)[3]	2.914	20	20	2
Dichlorofluoromethane	2.950	1	0.5	2
Trichlorofluoromethane	3.013	1	0.5	2
Ethanol	3.235	100	100	200
Ethyl ether (Diethyl ether)	3.351	5	5	10
1,1-Dichloroethene	3.364	1	0.5	2
Acrolein	3.488	50	50	20
1,1,2-Trichloro-1,2,2-trifluoroethane	3.615	1	0.5	2
Acetone	3.667	5	5	50
Iodomethane	3.783	10	10	20
Isopropyl alcohol	3.836	20	50	50
Carbon disulfide	3.857	1	0.5	2
Acetonitrile	4.016	20	20	20
Allyl chloride (3-Chloro-1-propene)	4.026	2	10	10
Methyl acetate	4.047	10	10	10
Methylene chloride	4.163	5	5	10
2-Methyl-2-propanol	4.311	10	10	50
Acrylonitrile	4.448	10	10	10
trans-1,2-Dichloroethene	4.480	1	0.5	2
MTBE	4.490	1	0.5	2
Hexane	4.807	2	0.5	10
1,1-Dichloroethane	4.976	1	0.5	2
3,3-Dimethyl-1-butanol	5.021	10	10	0
Vinyl acetate	5.050	10	10	20
Isopropyl ether (IPE, Di-isopropyl ether)	5.071	2	0.5	2
2-Chloro-1,3-butadiene (Chloropopene)	5.092	5	5	5
tert-Butyl ethyl ether (Ethyl-tert-butyl-ether, ETBE)[3]	5.514	1	0.5	5
2,2-Dichloropropane	5.683	1	0.5	2

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
cis-1,2-Dichloroethene	5.683	1	0.5	2
2-Butanone	5.715	50	50	50
Ethyl acetate	5.788	5	5	50
Propionitrile	5.788	10	10	50
Methacrylonitrile	5.968	20	20	50
Chlorobromomethane	5.978	1	0.5	2
Tetrahydrofuran	6.063	5	5	20
Chloroform	6.084	1	0.5	2
1,1,1-Trichloroethane	6.327	1	0.5	2
Cyclohexane	6.390	5	1	10
1,1-Dichloropropene	6.527	1	0.5	2
Carbon tetrachloride	6.538	1	0.5	2
Isobutyl alcohol	6.696	50	50	100
Benzene	6.801	1	0.5	2
1,2-Dichloroethane	6.823	1	0.5	2
tert-Amyl-methyl-ether (TAME)	6.960	1	0.5	2
n-Heptane	7.150	2	2	4
n-Butanol	7.582	100	20	100
Trichloroethene	7.656	1	0.5	2
Ethyl acrylate	7.815	5	5	10
1,2-Dichloropropane	7.973	1	0.5	2
Methylcyclohexane	7.973	5	0.5	10
Dibromomethane	8.131	1	0.5	2
Methyl methacrylate	8.142	5	5	10
1,4-Dioxane	8.184	200	200	200
Dichlorobromomethane	8.353	1	0.5	2
2-Nitropropane	8.680	5	5	10
2-Chloroethylvinyl ether	8.796	10	10	20
cis-1,3-Dichloropropene	9.007	1	0.5	2
4-Methyl-2-pentanone	9.239	5	5	50
Toluene	9.503	1	0.5	2
trans-1,3-Dichloropropene	9.830	1	0.5	2
Ethyl methacrylate	9.988	10	10	10
1,1,2-Trichloroethane	10.115	1	0.5	5
Tetrachloroethene	10.337	1	0.5	2
1,3-Dichloropropane	10.368	1	0.5	2
2-Hexanone	10.516	5	5	50
Chlorodibromomethane	10.727	1	0.5	2
n-Butyl acetate	10.738	10	10	40

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
Ethylene dibromide (EDB, 1,2-Dibromo-ethane)	10.907	1	0.5	2
Chlorobenzene	11.698	1	0.5	2
1,1,1,2-Tetrachloroethane	11.835	1	0.5	2
Ethylbenzene	11.888	1	0.5	2
m & p-Xylene	12.088	1	0.5	2
o-Xylene	12.743	1	0.5	2
Styrene	12.774	1	0.5	2
Bromoform	13.080	1	0.5	2
Isopropylbenzene (Cumene)	13.418	1	0.5	2
Cyclohexanone	13.555	50	50	50
Bromobenzene	13.914	1	0.5	2
1,1,2,2-Tetrachloroethane	13.925	1	0.5	2
1,2,3-Trichloropropane	13.988	1	0.5	2
trans-1,4-Dichloro-2-butene	14.030	5	5	10
n-Propylbenzene	14.115	1	0.5	2
2-Chlorotoluene	14.241	1	0.5	2
1,3,5-Trimethylbenzene	14.410	1	0.5	2
4-Chlorotoluene	14.421	1	0.5	2
tert-Butylbenzene	14.917	1	0.5	2
Pentachloroethane	14.927	5	5	10
1,2,4-Trimethylbenzene	14.990	1	0.5	2
1,3-Dichlorobenzene	15.391	1	0.5	2
4-Isopropyltoluene (p-Isopropyltoluene)	15.476	1	0.5	2
sec-Butylbenzene	15.524	1	0.5	2
1,4-Dichlorobenzene	15.529	1	0.5	2
1,2,3-Trimethylbenzene	15.613	1	0.5	2
Benzyl chloride	15.729	10	5	20
1,2-Dichlorobenzene	16.056	1	0.5	2
n-Butylbenzene	16.056	1	0.5	2
1,2-Dibromo-3-chloropropane	17.101	10	5	5
1,3,5-Trichlorobenzene	17.375	1	0.5	2
1,2,4-Trichlorobenzene	18.135	1	0.5	2
Hexachlorobutadiene	18.346	1	0.5	2
Naphthalene	18.431	5	5	5
1,2,3-Trichlorobenzene	18.716	1	0.5	2
2-Methylnaphthalene	19.729	10	5	5
INTERNAL STANDARDS/SURROGATES				
Dibromofluoromethane	6.284			
1,2-Dichloroethane-d_4	6.717			

Compound	Retention Time (minutes)	Typical Reporting Limits		
		Water Standard Level (µg/L)	Water Low-Level (µg/L)	Soil Wet Weight (µg/kg)
Fluorobenzene	7.160			
Toluene-d_8	9.408			
Chlorobenzene-d_5	11.656			
4-Bromofluorobenzene	13.671			
1,4-Dichlorobenzene-d_4	15.486			

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030 /NV05-107 (aqueous samples or Methanol extracts of bulk containers) and 5035 / NV05-108 (solid and waste oil samples), is used for volatile organic analytes.
1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor/Manager or the Technical Director. All abnormalities must be noted in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. The analytes are introduced directly to a capillary column for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) directly interfaced to the gas chromatograph (GC).
2.2 Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using at least a five-point calibration curve.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components are avoided, since such materials out-gas organic compounds which are concentrated in the trap during the purge operation. Analyses of blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, perform maintenance. **Subtracting blank values from sample results is not permitted**.
4.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. Re-analyze any suspect samples.
4.3 Special precautions are taken to analyze for Methylene chloride. The analytical and sample storage areas are isolated from all atmospheric sources of Methylene chloride. Otherwise, random background levels result. Since Methylene chloride permeates through PTFE tubing, all

gas chromatography carrier gas lines and purge gas plumbing is constructed from stainless steel or copper tubing.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly Methylene chloride and Fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank, prepared from organic-free reagent water and carried through the sampling, handling, and storage, serve as a check on such contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and should cool them to room temperature prior to working on them.
- The mass spectrometer is under vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- Kevlar gloves must be worn when opening and closing VOA vials.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure causes burn if not flushed with water. Causes mild irritation to skin. Prolonged exposure causes burn if not flushed with water.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors causes coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Trisodium phosphate		None listed	Keep in closed container; avoid high temperatures and strong acids.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Purge-and-trap device for aqueous samples at ambient temperature, described in Method 5030 / NV05-107.
- Purge-and-trap device for solid samples at 40°C, described in Method 5035 / NV05-108.
- The trap is VOCARB 3000 – 10.0-cm Carbopack™ B/6.0-cm Carboxin™ 1000/1.0-cm Carboxin 1001. The amount of thermal decomposition products formed must be routinely tracked by daily monitoring of the formation of Chloromethane and Bromomethane.
- Gas chromatography/mass spectrometer/data system
  - Gas chromatograph (HP): Analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

Injector temperature:	250°C
MS interface temperature:	260°C
Carrier gas (He) flow rate:	Constant flow of 1.0 mL/minute.
Initial temperature:	45°C hold for 6 minutes.
Temperature program:	13°C/minute to 150°C; 18°C/minute to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted (2 minutes)
Split ratio (min.)	1:10

May vary by instrument; see maintenance log for current program.

- The capillary column is directly coupled to the source.
- Gas chromatographic column: DB-624, 20 m x 0.18 mm with 1.0 µm film thickness, or equivalent.
- Mass spectrometer: Capable of scanning from 35 to 300 amu every 1 second or less using 70 volts (nominal) electron energy in the electron impact ionization mode. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.
- Data system (HP Chem Station with Enviroquant and CHROM): A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching the GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is used that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is also available.

6.2 Supplies

- Microsyringes, 10, 25, 100, 250, 500, and 1,000 µL.
- Syringes, 5, 10, or 25 mL.
- Balance, analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.
- Glass scintillation vials, 20 mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.
- Disposable pipets, Pasteur.
- Volumetric flasks, Class A, 10 mL, 50 mL and 100 mL, with ground-glass stoppers.
- Spatula, stainless steel, or wooden tongue depressor.
- Helium for carrier gas.
- Nitrogen for purge-and-trap gas.
- Narrow-range pH paper.
- Residual chlorine test strips.
- Sea or Ottawa sand for blank and LCS soil matrix.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are generally used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. See the QA Manual and SOP Reagent and Standard Purchase / NV08-214 for more information on reagent chemicals, such as shelf-life and storage.

7.2 Reagent water, analyte-free.

7.3 Methanol, CH_3OH: Purge-and-trap grade or equivalent, demonstrated to be free of analytes at the MDL. Store apart from other solvents.

7.4 Hydrochloric acid (1:1 v/v), HCl: Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

7.5 Stock solutions: Stock solutions are prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in Methanol, using assayed liquids. Any specific standards or procedure for making standards mentioned in this SOP may be substituted with equivalent standards or procedures. See standard log for specific standard information.

7.5.1 **Primary Standards**

		For Working Standard		
Name of standard	**Vendor[2]/Conc (µg/mL)**	**Volume used (mL)**	**Final Volume (mL)**	**Conc. (µg/mL)**
Full List Non-gas Standard				
Custom 8260 VOC mega-mix[1] without gases	Restek 567641/2000, 4000, 10000,-20000, 40000	2.5	50	100 – 2000
Megamix additions	Restek 567647/2000, 4000,20000, 50000, 100000	2.5	50	100-5000
Ketones	Restek 567642/10000	2.5	50	500
Acrolein	Restek 567644 /5000	2.5	50	250
Cyclohexanone	567648/20000	2.5	50	1000
Vinyl acetate	Restek 567646/4000	2.5	50	200
2-Chloroethyl vinyl ether	Restek 567643 /2000	2.5	50	100
List 2: Pentachloroethane, 2-Methylnaphthalene	Restek 567719 / 2000	2.5	50	100

		For Working Standard		
Name of standard	**Vendor[2]/Conc (µg/mL)**	**Volume used (mL)**	**Final Volume (mL)**	**Conc. (µg/mL)**
1-Methylnaphthalene	Restek 31283/1000	0.1	2	50
Full List Gas Standard				
Gas Mix	Restek 567645/2000	1.0	20	100
Short List				
Short List Mix	Ultra CUS-7011/100-1000	5	10	50-500
3,3-Dimethyl-1-butanol	Restek 563892/20000	0.25	10	500
1 Custom 8260 VOC mix has variable concentrations. See the standard log for exact compound concentrations.				
2 The vendors/catalog numbers are recommended; equivalent products are acceptable.				

7.5.1.1 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at ≤ 6°C or less or as recommended by the standard manufacturer. Return standards to storage as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of target compounds.

7.5.1.2 **Frequency of Standard Preparation**

7.5.1.2.1 Monitor standards for the permanent gases frequently by comparison to the initial calibration curve. Prepare fresh standards if this check exceeds a 20% drift. **Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented**. Dichlorodifluoromethane and Chloromethane are usually the first compounds to evaporate from the standard and, therefore, are to be monitored very closely when standards are held beyond one week.

7.5.1.2.2 Monitor standards for the non-gases frequently by comparison to the initial calibration. Prepare fresh standards if this check exceeds a 20% drift. Undiluted standards for non-gases usually need to be replaced after **one month for working standards and three months for opened stock standard** or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-Chloroethyl vinyl ether and Styrene may need to be prepared more frequently.

7.5.1.3 **Secondary dilution standards:** Using stock standard solutions, prepare secondary dilution standards in Methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards are stored with minimal headspace and, except for gases, are good for 2-4 weeks unless acceptability is demonstrated. Replace secondary standards for gases after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. Handle and store standards as stated above and return them to the refrigerator or freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

7.5.1.3.1 The working calibration standard for the Non-Gas mixture is made by adding 2.5 mL of each of the first six standards in the Primary Standard table above in 50.0 mL Methanol in a Class A volumetric. The Gas

Standard is added right before use as described in the calibration section.

7.5.2 **Internal Standard/Surrogate Standard Mix (IS/SS)**

7.5.2.1 The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. Prepare internal standard stock and secondary dilution standards in Methanol. Stock standard is 250 µg/mL, Restek 567649, or equivalent.

7.5.2.2 The surrogates are Toluene-d_8, 4-Bromofluorobenzene (the GC/MS Tuning Standard), 1, 2-Dichloroethane-d_4, and Dibromofluoromethane. Stock standard is 2500 µg/mL, Restek 567650, or equivalent.

7.5.2.3 Prepare a 250 µg/mL IS/SS standard by diluting 5.0 mL of stock internal standard (250 µg/mL) and 5.0 mL stock surrogate standard (2500 µg/mL) to a final volume of 50.0 mL of Methanol in a Class A volumetric.

7.5.3 **Bromoform Breakdown Check:** Purchase 50 g neat Bromoform from Sigma-Aldrich 241032-50G, or equivalent.

7.5.3.1 Prepare a 20 µg/L standard by adding 0.02 g of the neat Bromoform standard to 1000 mL reagent water.

7.5.4 **Second-Source Standards for Initial Calibration Standard (ICV):** The ICV is a **second-source** standard that contains all the 8260 compounds. Prepare as for the primary standard with the only difference being that the vendor numbers have a ".sec" on the end of the number.

7.6 Sodium bisulfate or Trisodium phosphate for soil sample preservation. See SOP 5035 / NV05-108.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time From Collection to Analysis	Reference
Water[2]	3 x 40-mL VOAs (Optional: TSP)	40 mL	pH < 2 with Hydrochloric acid. Cool 0-6°C, No headspace. Keep in dark. If Chlorine residual present, add 0.008% $Na_2S_2O_3$.	14 days, 7 days if not acidified.	SW846 Chapters 2 and 4
Low-concentration Soil	2 pre-weighed vials, stirring bar	5 g	0-6°C, 5 mL preservative[1]		
	2 EnCores™		0-6°C, Add 5 g sample and 5 mL preservative to pre-weighed vial with stirring bar within 48 hours of collection		
High concentration Soil	2-oz. glass[3] or 25 g Encore™	5g or 25 g	0-6°C, Add 1 mL Methanol/gram soil	Transfer to VOA within 48 hours, then 14 days	

[1]0.2 gram sodium bisulfate or Trisodium phosphate/mL reagent water

[2]2-Chloroethyl vinyl ether degrades in acid-preserved samples; its analysis requires a non-preserved vial. If analyzing a sample for combined purgeable halocarbons, aromatics, Acrolein, and Acrylonitrile, analyze the sample within 7 days. Alternatively, collect at least 2 separate vials for analysis: one vial preserved to pH 4-5 with HCl for Acrolein and Acrylonitrile, and a second vial for the other analytes preserved to pH <2 with HCl.

[3]See SOPs 5030 / NV05-107 for waters and 5035 / NV05-108 for soils/solids, including the soil freezing option, with or without water.

Analysis Method	Sample Storage	Holding Times from Date and Time of Collection			
		MeOH Addition	Shipping	Extraction	Analysis
Wisconsin VOC Soils	VOC vial	Immediately	4 days	21 days	21 days
	Brass Tube	within 2 hours	4 days	21 days	21 days
	EnCore™	within 48 hours	40 hours	21 days	21 days

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC: The following QC is run **every batch of no more than 20 samples:**

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Method blank	One per analytical prep batch and after calibration (see Section 9.2)	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank. If target > 10x blank, report but qualify.
LCS[4] for all analytes using the primary standard.	One[6] per prep batch	See LIMS[5]	Re-prep[3] and analyze the LCS and all samples in the affected analytical batch. If high and samples are ND, report. If low, re-prep. If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prep and re-analyze the batch. If the LCS exceeds the upper control limit AND the sample from that batch is less than the RL, the data is acceptable to report.
MS/MSD using the primary standard	One per batch per matrix, if insufficient sample for MS/MSD, then analyze a LCS/LCSD.	See LIMS	None (LCS is used to determine if data is acceptable).
Surrogate	Every sample, spike, standard, and blank.	See LIMS	Check system, re-analyze, re-prep[3], may qualify. If %recovery is high and the sample is ND, it is acceptable to report. If low, re-prep and rerun. If the surrogate % recovery exceeds the upper control limit AND a sample is greater than the RL, re-prepare and re-analyze the sample. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prepare the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed
pH check	All water samples.	pH ≤2or ≥ 11	If the pH is > 2 but less than 11, comment the data and LIMS.
Residual chlorine check (North Carolina samples only)	Each sample.	Residual chlorine must be negative.	If the residual chlorine is positive, then comment the data, and LIMS.

[1]This is a summary of the acceptance criteria.
[2]All abnormalities must be noted in LIMS.
[3]If unable to re-prep the samples because of insufficient sample volume or holding time has expired, place a comment in LIMS.

[4] All AZ, MA, TX, and WV samples require a LCS duplicate in each batch.
[5].See Section 16 for South Carolina LCS acceptance criteria and Minnesota Ethanol acceptance criteria.

- A **Method Blank** is run with each analytical batch. The blank is carried through all stages of the sample preparation and measurement using the appropriate blank matrix (reagent water or sand).
- A **Laboratory Control Sample (LCS)** is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix (reagent water or sand) similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes from the primary source.

Matrix	LCS Preparation	Final Concentration
Water	Add 50 µL of the primary source standard to 50.0 mL reagent water in a Class A volumetric flask.	50 – 5000 µg/L
Low-concentration Soil	Add 5 µL of the primary source standard to a VOA vial containing 5.0 g sand and 5 mL preservative and a stirring bar.	50 - 5000 µg/kg
High-concentration Soil (analyzed as waters)	Add 50 µL of the primary source standard to 50.0 mL reagent water in a Class A volumetric flask.	50 - 5000 µg/kg

- **Matrix Spike/Matrix Spike Duplicate:** Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair for each batch.

Matrix	MS/MSD Preparation	Final Concentration
Water Batch	Add 43 µL of the primary source standard to the client's sample in VOA vials.	50 – 5000 µg/L
Low-concentration Soil Batch	Add 5 µL of the primary source standard to a VOA vial containing 5 g preserved client sample (with stirring bars).	50 - 5000 µg/kg
High-concentration Soil Batch (analyzed as waters)	Add 1.0 mL of the Methanol-extract-of-client-sample and 50 µL of the primary source standard and dilute with reagent water in a 50-mL, Class A volumetric.	50 - 5000 µg/kg

- **Surrogate standards**: The analyst monitors both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, QA/QC standard, and blank with surrogate compounds which are not expected to be affected by method interferences. The surrogate and internal standards are prepared together as described in Section 7.
  - The IS/SS standard mix (250 µg/mL each) is added by the autosampler (nominally 1 µL) during all analyses with the exception of the calibration.

Purge Volume, mL	Concentration of IS/SS Standards in Sample, µg/L
5	50
10	25

- **pH Check:** The analyst must document that each sample has a pH ≤ 2 or ≥ 11 by checking with narrow-range pH paper. The pH check is performed after sample analysis to avoid

contamination and creation of a headspace in the sample vials. Record as pH <2 or >2 or > 11.

- **Residual Chlorine Check:** The analyst must document the presence/absence of residual chlorine in North Carolina samples by checking with residual chlorine test strips.

9.2 Instrument QC

Italicized information is unique to 8260C.

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
a. Check of mass spectral ion intensities, i. e., BFB Tune	Prior to initial calibra-tion or Continuing calibration verification, every 12 hours.	Refer to criteria for Tune criteria	Retune the instrument and verify (instrument maintenance may be needed).
b. Bromoform Break-down Check	At beginning of daily sequence.	≤ 0.5 µg/L Bromomethane; ≤ 0.5 µg/L Chloromethane	Re-condition or replace trap. Re-calibrate.
Minimal five-point initial calibration for all target analytes. Single-point surrogate calibration	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8260B: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%. $r^2 \geq 0.990$ or $r \geq 0.995$. Re-calculate low point; must be within 30% true. *8260C: Minimum RF for initial and continuing calibration varies by analyte (see Calibration standards below). RSD ≤ 20% each target.* $r^2 \geq 0.990$ or $r \geq 0.995$. *Up to 10% of targets may exceed these criteria. If using linear regression, re-fit lowest calibration point. It must be ± 30% or re-calculate.*	Correct problem then repeat initial calibration.
Initial calibration verification (ICV), must be from a second source	Immediately following each initial calibration.	All analytes within 30% of expected value. Problematic compounds may be within 40%.	Correct problem then repeat initial calibration. ICV must be run prior to reporting samples.
Continuing Calibration Verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	8260B: CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). SPCCs: minimum RF. All other target compounds ≤ 30%, except for specific compounds which may have a % difference ≤ 40%. *8260C: All targets of interest ≤ 20%. Up to 20% of targets may exceed this criterion. Common targets meet minimum RF.*	Correct problem then repeat CCV (re-calibrate if necessary) and re-analyze any samples processed with that CCV. If the CCV is high and the sample is ND, it is acceptable to report.[3]
Continuing Calibration Blank	After each CCV.	< ½ RL or MDL, whichever is greater.	Correct problem, repeat.
Internal Standards[3]	Every sample, standard and blank.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of most recent ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning.
Retention time window calculated for	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then re-analyze all samples analyzed since the last retention time

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
each analyte			check.
MDL verification (MDLV)	Minimum yearly.	Detectible.	Re-evaluate MDL standard used and MDL; see Technical Director.

[1] This is a summary of the acceptance criteria.
[2] All abnormalities must be noted in LIMS.
[3]Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.

- **BFB Tuning and Breakdown Check**:
  - **BFB Tuning**: At the beginning of each 12-hour analytical shift and **prior** to the analysis of samples or calibration standards, inject 50 ηg or less of the 4-Bromofluorobenzene standard into the GC/MS system (1 µL of 250 µg/mL standard /50 mL reagent water, purged at a 1:10 split for a 25 ηg on the column). (BFB is one of the surrogate compounds.) The resultant mass spectra for the BFB **must** meet the tuning criteria below before sample analysis begins.

BFB (4-Bromofluorobenzene) Mass Intensity Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101 % of m/z 174
177	5 to 9% of m/z 176

  - Three options are available for acquiring the spectra for reference to meet the BFB tuning requirements:

Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex ± 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct.

Option 2 The scan across the peak at one half peak height may be averaged and background-corrected.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is still required.

Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance.**

- Note: All subsequent standards, samples, controls, and blanks associated with a BFB tune **must** use identical mass spectrometer instrument conditions.

- **Bromoform Breakdown Check**: The daily BFB Tune/Breakdown Check containing surrogates, internal standards, BFB, and 20 µg/L Bromoform must be analyzed prior to the analysis of the Continuing Calibration Verification (CCV). If levels of Chloromethane or Bromomethane exceed 0.5 µg/L, then the trap may be too contaminated with salts or tightly bound contamination for analysis to continue. The trap must be replaced, and the system re-calibrated.

- **Calibration standards**: See Section 10.2.

SPCCs and CCCs are unique to 8260B. ***Italicized text is unique to 8260C.***

- **Initial System Performance Check Compounds (SPCCs)**: A system performance check is made before the calibration curve is used. Five compounds (the System Performance Check Compounds) are checked for a minimum average response factor, compound instability, and degradation caused by contaminated lines or active sites in the system. These compounds are Chloromethane, 1,1-Dichloroethane, Bromoform, Chlorobenzene, And 1,1,2,2-Tetrachloroethane. The minimum mean response factors for the volatile SPCCs must be met and are as follows:

Chloromethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
1,1-Dichloroethane	0.10
Chlorobenzene	0.30

Example problems include:

- Chloromethane is the most likely compound to be lost if the purge flow is too fast.
- Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve Bromoform response.
- Tetrachloroethane and 1,1-Dichloroethane are degraded by contaminated transfer lines or active sites in trapping materials.

- **Initial Calibration check compounds (CCCs):** The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites in the system. Meeting the CCC criteria is **not** a substitute for successful calibration of the target analytes. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

- Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for **all** target analytes from the initial calibration with the equations in Section 11.
- The RSD must be less than or equal to **15% for each target analyte**; however, the

RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak or contamination and/or column reactive sites is necessary before re-attempting calibration. The CCCs may not be in the project target list. If that is the case, each target must have a RSD < 15% or a correlation coefficient $r \geq 0.995$ ($r^2 \geq 0.990$) as calculated by the equations in Section 11. When using linear regression, re-calculate the low calibration point. It must be within 30% true.

- *For 8260C, the minimum RF for initial and continuing calibration is:*

RF	*For These Compounds*
0.1	*Critical compounds*
0.2	*1,1-Dichloroethane, Chloroform, Trichlorethene, Bromodichloromethane, cis-1,3-Dichloropropene, Tetrachloroethane, and 1,2,4-Trichlorobenzene*
0.3	*o-Xylene, Styrene, 1,1,2,2-Trichloroethane*
0.4	*Toluene, 1,2-Dichlorobenzene*
0.5	*Benzene, Chlorobenzene, 1,4-Dichlorobenzene*
0.6	*1,3-Dichlorobenzene*

  - *For 8260C, the must be less than or equal to 20% for each target analyte with up to 10% of compounds meeting the 40% criterion.*

- **Initial Calibration Verification (ICV)** is verified immediately after calibration using the introduction technique used for samples. Analyze a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS.
  - The ICV is made from the **second-source** standards, one at a time, as needed, to be run after an initial calibration: Add 25 µL of the second-source, non-gas, working standard to 50 mL reagent water in a 50-mL, Class A volumetric. Add 25 µL of the Gas Mix working standard to the Class A volumetric for a final concentration of 50-5000 µg/L.
  - The ICV of each target must be within 30% of the expected value, with the exception of the following poor purge efficiency analytes that may be within 40% of the expected value for up to 20% of targets:

Acrolein	Ethanol	2-Methylnaphthalene
t-Amyl alcohol (TAA)	t-Butyl formate (TBF)	Vinyl acetate
t-Butyl alcohol (TBA)	1-Methylnaphthalene	

  - If ICV criterion is not met, correct the problem and re-calibrate.
- **Continuing Calibration Verification (CCV):** Run every 12 hours of sample analysis, CCVs are often made each day for several instruments in the following proportions, always from the **primary** calibration standards:
  - Add 25 µL of the working calibration Non-Gas Standard and 25 µL of the primary Gas Mix to a 50-mL, Class A volumetric and dilute to the mark with reagent water. The final concentration is 50-5000 µg/L.
  - Area counts of the internal standards must be between 50 - 100% of the areas of the internal standards in the mid-point calibration standard. If not, inspect the GCMS for possible maintenance issues and then re-analyze. Contact the department supervisor for assistance in determining the appropriate course of action. **Do not report data from a failing internal standard associated with target compounds.**
  - **For 8260B Only: Continuing System Performance Check Compounds (SPCCs)**
    - A System Performance Check **must** be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard **must** meet its minimum

response factor. This is the same check that is applied during the initial calibration.
    - If the minimum response factors are not met, the system **must** be evaluated, and corrective action **must** be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.
- **For 8260B Only: Continuing Calibration Check Compounds (CCCs)**
    - After the system performance check is met, the CCCs are used to check the validity of the initial calibration, if present in the target list. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. See Section 11 for equations.
    - If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift), for any one CCC, then corrective action **must** be taken prior to the analysis of samples. **If the CCCs are not included in the list of analytes for a project and therefore not processed in the calibration standards, then all analytes must meet the 20% difference or drift criterion.**
    - Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC or target criteria **must** be met before sample analysis begins.
- **For 8260B Only: Continuing Evaluation of Non CCC/SPCC compounds** – The percent difference or drift for each of the non-CCC analytes is less than or equal to 30%. Recovery for some compounds with poor purge efficiency may exceed this 30% requirement and still be deemed acceptable provided all of the following criteria are met:
    - Poor performing analytes are one of the following: Acrolein, tert-Amyl alcohol (TAA), tert-Butyl alcohol (TBA), tert-Butyl formate (TBF), Ethanol, 2-Methylnaphthalene, Vinyl acetate.
    - The percent difference or drift is less than or equal to 40%.
- *For 8260C, see ICAL and ICV information.*
- **Continuing Calibration Blank (CCB):** The CCB is reagent water or sand.
- **Internal Standards** are used to evaluate the effect of the sample matrix. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected.
    - The retention times of the internal standards in the calibration verification standard are evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Note any maintenance in the logbook.
    - Internal standards permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Attachment 1). If interferences are noted, use the next most intense ion as the quantitation ion.
    - **Internal standard response** - If the EICP area for any of the internal standards in the calibration verification standard and samples changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made.

When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Note any maintenance in the logbook.

- The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
  - If the internal standard is within criteria, report the second analysis.
  - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
  - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
- See Attachment 2 for the analytes corresponding to each internal standard.

- **Retention time windows**: Target analytes are identified on the basis of retention time windows.
  - Before establishing retention time windows, make sure that the chromatographic system is functioning reliably and that the operating parameters have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.
  - Establish the retention time windows for target analytes.
  - The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.
- **Method Detection Limit Verification (MDLV):** Annually verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	VOA vial
Low-concentration Soil	5 grams
High-concentration Soil	1 mL Methanol extract of soil

- All samples and standard solutions are allowed to warm to ambient temperature before analysis.
- Refer to SOP 5030 / NV05-107 for waters and 5035 / NV05-108 for soils/solids.
- For Wisconsin VOC soils, the following procedure must be performed for Methanol extraction of Soil/Sediment:

1	Hand-shake the sample in its vial containing Methanol vigorously for 2 minutes. Sonicate for 20 minutes.
2	Allow sediment to settle until a layer of Methanol is apparent.
3	Withdraw an appropriate aliquot of the Methanol extract for sparging and add to a VOA vial.
4	Analyze all reagent blanks and QC samples on the same instrument as that used for the samples.
5	If the responses exceed the calibration or linear range of the systems, use a smaller aliquot of Methanol extract or dilute the aqueous sample.

10.2 Initial Calibration: Refer to SOP Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002 and Calibration Curves (General) / CA-Q-S-005. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1 Evaluate the **BFB tuning criteria.**

2 Prepare the **Initial calibration standards** at a minimum of five different concentrations from the secondary dilution of stock standards or from a premixed certified solution in organic-free reagent water. **At least one of the calibration standards corresponds to a concentration at or below the laboratory reporting limit.** This low standard must have valid ion abundances for all monitored ions. The remaining standards define the working range of the system. Initial calibration standards are mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

Initial Calibration (5-point)

Primary Working Standard	Final Volume (mL)	Concentration (µg/L)
1	100	0.5-2.5
2	100	1 - 5
4	100	2 - 10
20	100	10 - 50
40	100	20 - 100
100	100	50 - 250
200	100	100 - 500
400	100	200 - 1000

1 µL of IS/SS Standard at 250 µg/mL is added by the autosampler to 5 mL for a 50-µg/L concentration in the standards and samples with a 5-mL purge; 1 µL/10 mL for a 25 µg/L concentration. See Section 9.1 for make-up and final concentrations for other purge volumes. The surrogate calibration is a single-point.

- All target analytes for a particular analysis must be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes for which the method has been demonstrated. **However, the laboratory must not report a quantitative result for a target analyte that was not included in the calibration standard(s).**
- **Internal Standards:** The calibration standards must also contain the internal standards chosen for the analysis. Calibration standards for soils must also contain the preservative Na_2SO_4. See Method 5035 / NV05-108 for how to accomplish the preservation.
- **Surrogates**: Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. With improvements in instrumentation and more reliance on the autosampler, an option is available allowing the autosampler to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option, the surrogate standards in the initial calibration can be averaged to develop a response factor and an effective one-point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option, the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout, and the recovery of this known concentration can easily be attained without demonstrating if the response is linear.
- **Technique:** To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a Class A, volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection and stopper. Mix by inverting the flask three times. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and are prepared daily. Transfer each standard to separate VOA vials.

	• **Water or Soil Samples:** A different calibration curve is necessary for Methods 5030 / NV05-107 and 5035 / NV05-108. Calibration **must** be performed using the same sample introduction technique that is used for samples. For Method 5030, the purging efficiency varies with purge volume; therefore, develop the standard curve with whichever volume of sample that is to be analyzed.
3	**Calibration sequence**: 1 BFB Tuning Criteria 2 ICAL 3 ICV 4 ICB
4	**Tabulate the area response of the characteristic ions** (see Attachment 1) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards.
5	• Evaluate the RSD or linearity.
6	For 8260B: evaluate the SPCC and CCC compounds for the initial calibration criteria. For 8260C: evaluate each target.
7	Evaluate the retention times and minimum response factors.
8	Evaluate the success of the initial calibration by immediately running an Initial Calibration Verification (ICV).
9	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.

10.3 Daily GC/MS Calibration Verification

1	Evaluate the BFB tuning and Breakdown Check criteria.
2	Evaluate the CCV and CCB.
3	For 8260B, evaluate the SPCC and CCC compounds for the continuing calibration criteria. For 8260C: evaluate each target.

10.4 Example Analysis Queue / Sequence (based on 12 hours)

1	Tune/Breakdown Check
2	CCV, for daily and ongoing calibration check
3	LCS
4	Blank
5	Samples
6	Matrix Spike
7	Matrix Spike Duplicate
When 12 hours have passed, run a 2nd tune and CCV before running more samples, no more than 20 samples in a 12-hour batch.	

- **Dilutions**: If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the upper calibration standard, the sample must be reanalyzed at a higher dilution. **Secondary ion quantitation is allowed only when there are sample interferences with the primary ion**.
  - When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free, reagent water blank or the repeating of suspected samples. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is

demonstrated to be free of interferences. Repeat all affected samples.

- Prepare dilutions such that the response of the major constituents (previously saturated peaks) is in the upper half of the linear range of the curve.
- The following procedure is used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays.

1	Dilutions are made in Class A, volumetric flasks (50 to 100 mL). Select the volumetric flask that allows for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.
2	Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.
3	Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, three times. Repeat above procedure for additional dilutions.
4	Fill a VOA vial with the diluted sample and cap.

- For high concentration samples, see SOP 5035 / NV05-108.

10.5 Qualitative analysis

The qualitative identification of each compound determined by this method is based on relative retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. See Attachment 1 for primary and secondary ions for each compound. Compounds are identified as present when the following criteria are met:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree with 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the ± 30 % relative intensity, report the compound as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair.
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i. e., only one

chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum might contain extraneous ions contributed by the co-eluting compound. If all of the ions associate with the reference spectrum for the target analyte are present and within the ± 30% criteria, a positive result must be assumed even in the presence of extraneous ion fragments without presumptive evidence for a negative identification. (All ions associated with the target analyte are also present in the interfering peak.) The analyst must carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. Add appropriate qualifiers in Element (ID2).

- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.
- For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification is determined by the purpose of the analyses being conducted. Data system library search routines are not to use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
- For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:
  - Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample spectrum.
  - The relative intensities of the major ions must agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
  - Molecular ions present in the reference spectrum must be present in the sample spectrum.
  - Review ions present in the sample spectrum but not in the reference spectrum for possible background contamination or presence of co-eluting compounds.
  - Review ions present in the reference spectrum but not in the sample spectrum for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the **primary** characteristic ion. The internal standard used is the one nearest the retention time of that of a given analyte. See Attachment 1.
- If the RSD of a compound's response factors is 15% or less, then the concentration is determined using the average response factor (*RF*) from initial calibration data.
- Where applicable, the concentration of any non-target analyte identified in the sample may be estimated. The same formulae are used with the following modifications: The areas A_x and A_{is} are from the total ion chromatograms, and the RF for the compound must be assumed to be 1.
- The resulting concentration is reported indicating:
  - that the value is an estimate, and

- in Level 4 data packages, which internal standard was used to determine the concentration. Use the nearest internal standard free of interferences.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\% \text{ Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard

11.4 Standard Deviation, Relative Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - RF_{mean}\right)^2}{n-1}} \qquad RSD = \frac{SD \times 100}{RF_{mean}}$$

RF_i = RF for each of the calibration standards
RF_{mean} = mean RF for each compound from the initial calibration
n = Number of calibration standards, e. g., 5

11.5 % Difference, % Drift

$$\% \text{ Difference} = \frac{(RF_v) - (\text{Avg. RF}) \times 100}{\text{(Avg. RF)}}$$

RF_v = RF from verification standard
Avg. RF = Average RF from Initial Calibration.

$$\% \text{ Drift} = \frac{\text{Result - True Value} \times 100}{\text{True Value}}$$

11.6 Linear Calibration Using a Least Squares Regression: A linear calibration model based on a least squares regression may only be employed if RSD does not meet the acceptance criteria.

For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

by minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y_i', the response calculated from the constructed equation). The regression equation is:

$y_i' = ax_i + b$

a = regression coefficient or the slope of the line.
b = the y-intercept.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
x_I = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(x_i - x_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i(x_i - x_i')^2$$

w_i = weighting factor for the i^{th} calibration standard (w=1 for unweighted least squares regression).
x_i – observed instrument response (area) for the i^{th} calibration standard.
x_i' = predicted (or calculated) response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used.

Examples of allowed weighting factors which place more emphasis on numbers of smaller value are:

$w_i - 1/x_i$ or $w_i = 1/x_i^2$

Do not include the origin (0, 0) as an extra calibration point. The use of a linear regression may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. If it is necessary to report results at lower concentrations, then the analyst must run a calibration that reaches those lower concentrations.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.990$.

11.7 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response ratio
x = Concentration

11.8 Concentration Calculation

Concentration = (µg/L from instrument) (dilution factor)

12.0 <u>Method Performance</u>

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Aqueous waste generated from analysis may have a pH of less than 2.0. Transfer to waste disposal for neutralization and then dump into the sanitary sewer.
- Solid waste generated from analysis is placed in the trash.

15.0 References / Cross References

15.1 EPA Method 8260B, SW-846 Update III, Revision 2, December 1999, **Method 8260C**, Update.IV, Rev. 3, August 2006.

15.2 Method 8000B, SW-846, Revision 2, December 1996, **Method 8000C**, Revision 3, March 2003.

15.3 Method TPH-GRO by Method 8260B, MRBCA (Missouri) Guidance Document, Final Draft, February 24, 2004.

15.4 California GRO, CA LUFT 8015.

15.5 TestAmerica Nashville's Quality Assurance Manual.

15.6 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.7 SOPs: Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002, Calibration Curves (General) / CA-Q-S-005, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, Sample Homogenization, Sub-sampling & Compositing / NV08-229, 5030 / NV05-107, 5035 / NV05-108.

15.8 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

State	Modification
Ohio specific criteria	Only those compounds in EPA Method 8260B may be reported (superscript 1 in the table in Section 1.1). Some compounds in this SOP are not part of the original 8260B method. The method blank must be less than the RL for Ohio samples. See SOP 8260/NVOH05-77.
Missouri GRO	Prepare 1:1 mixture of unleaded gasoline and #2 diesel fuel in Methanol as GRO is defined by setting retention time window from 0.1 minutes before C_6 to 0.1 minutes after C_{10}. Verify RT window with the standard daily (every 24 hours).
California	California LUFT GRO uses gasoline and the retention time window of C_4 (t-Butanol) to

GRO	C_{12}.
Michigan GRO	See the Michigan GRO requirements below.
South Carolina	See the special LCS acceptance criteria and special state PQL requirements below.
Minnesota	See the special Ethanol analysis requirements below for water samples.

TPH-GRO by Method 8260B

Standards

- C4-C12 Standards
  - Primary: Restek 30096, 5000 µg/mL, or equivalent.
  - Secondary: O2SI 020246-S6, 10,000 µg/mL, or equivalent.
- C6-C10 Standards
  - Primary: Restek 31484, 20,000 µg/mL, or equivalent.
  - Secondary: Ultra CUS-8324, 10,000 µg/mL, or equivalent.

Sample Introduction

- Samples are purged onto the GC/MS system using all protocols specified in SW-846 Method 5030 and 5035.
- Surrogates and internal standards specified by Method 8260B are added to water and soil samples prior to purging.

Sample Analysis

- The BC/MS system is tuned to BFB tune criteria listed in Method 8260B at the frequency specified in Method 8260B.
- A 5-point standard curve is used to quantitate TPH-GRO by the internal standard technique.
- For **Missouri** GRO, the stock standard solution is a mixture of unleaded gasoline and Number 2 diesel fuel.
- For **Californi**a GRO, the stock standard is unleaded gasoline.
- The lowest calibration standard should be at or below the reporting limit of the method.
- For **Missouri,** retention time windows are defined for TPH-GRO by analyzing a standard containing C_6 and C_{10}. The retention time window is defined as $>C_6$ to C_{10}. The standard containing C_6 and C_{10} must be analyzed every day samples are analyzed in order to verify that the retention time windows are constant.
- For **California**, the retention time window are defined as $>C_4$ to C_{12}.
- For **Michigan**,
  - Use unleaded gasoline for calibration.
  - The retention time window is defined as C_6 (hexane) to C_{10} (n-decane).
  - The holding time for water and soil is 14 days.
  - For soil preparation, shake the Methanol and sample for 2 minutes, then sonicate in a water bath for 20 minutes.
  - For oil samples, add 2 g sample to 40 mL Methanol; wait 24 hours before analysis.
  - Use the internal calibration technique, summing the range.
  - Use only linear regression; $r \geq 0.990$, $r^2 \geq 0.981$.
  - ICV and CCV must be ± 20% true.
- Because the retention time window is several minutes wide for TPH-GRO, the GC/MS data system may not accurately or appropriately establish the proper baseline for calibration or quantitation. The analyst **must** visually examine the computer-generated baseline for every analytical run and manually adjust the baseline when needed. A properly drawn baseline must extend over the entire retention time window and include

the area under the entire TPH-GRO series of peaks. It is not appropriate to draw the baseline "peak to peak."

- The total ion chromatogram (TIC) **must** be used to calculate the area under the peak for TPH-GRO calibration and quantitation determinations over the entire retention time window.
- Area counts for the internal standards and surrogates added during sample preparation **must** be subtracted from the total area count for TPH-GRO. This is accomplished by subtracting the area count of the method blank from all subsequent calibration and analytical runs.
- The %RSD for the calibration curve for TPH-GRO must be less than or equal to 20%, so that linearity through the origin can be assumed and an average calibration factor used for calculations.
- A continuing calibration verification standard (CCV) must be analyzed at the beginning of each batch. The standard concentration should be at the mid-point of the calibration curve. If the %RSD exceeds 20%, a new curve must be generated.
- A method blank must be analyzed once per day to insure the analytical system is free of background contamination.

South Carolina LCS Acceptance Criteria and Special State PQL Requirements

- All routinely reported analytes require 70-130% LCS recovery. The following exceptions of poor-performing analytes require 60-140% LCS recovery:

Acetonitrile	Dichlorodifluoromethane
Acrolein	2,2-Dichloropropane
Acrylonitrile	3,3-Dimethyl-1-butanol
t-Amyl alcohol	1,4-Dioxane
Bromomethane	Ethanol
t-Butyl alcohol	2-Hexanone
t-Butyl formate	Isopropyl alcohol
Chloroethane	4-Methyl-2-pentanone
Chloromethane	Vinyl chloride
1,2-Dibromo-3-chloropropane	

- Instrumentation used for South Carolina samples must be able to achieve and report the following South Carolina PQLs when those compounds are requested:
  - Acrolein 5 ug/L
  - Acrylonitrile 5 ug/L
  - 2-Chloroethyl vinyl ether 5 ug/L
  - Methylene chloride 2 ug/L

Minnesota Ethanol Analysis Requirements for Water Samples

- The calibration standard used for Ethanol must be a water-based standard and not a Methanol-based standard. Ethanol water-based standards must be stored at <4°C.
- Initial calibration: The recovery (accuracy) for each point in the curve must be 70-130% except for the lowest point in the curve which must be 60-140%.
- Continuing calibration verification: Analyze one low-level Ethanol standard at the report level (RL) and one mid-level Ethanol calibration verification standard at approximately 500 µg/L prior to the samples. %R for Ethanol in the low-level standard must be 60-140% of the true

value. %R for Ethanol in the mid-level standards must be 70-130% of the true value and a % difference of ≤ 30%.

- For samples, absolute areas of the quantitation ions for the internal standard and surrogate must not decrease by more than 50% from the initial calibration.
- %R for Ethanol for the MS/MSD must be 70-130% with a relative percent difference (RPD) of ≤30%. %R for the LCS/LCSD must be 70-130% with a RPD ≤ 30%.
- The quantitation ion for Ethanol is 45 atomic mass units (AMU). Confirmation ions are 46 and 47 AMU. Ethanol standards must be analyzed separately from the normal VOC list due to the interference from Ethyl ether.

17.0 Attachments

17.1 Attachment 1, Characteristic Masses (m/z) for Purgeable Organic Compounds.

17.2 Attachment 2, Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation.

Attachment 1, Characteristic Masses (m/z) for Purgeable Organic Compounds

Compound	Primary Characteristic Ion	Secondary Characteristic Ion
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,1-Trichloroethane	97	99, 61
1,1,2,2-Tetrachloroethane	83	131, 85
1,1,2-Trichloro-1,2,2-trifluoroethane	101	151, 103, 153
1,1,2-Trichloroethane	97	83, 85
1,1-Dichloroethane	63	65, 83
1,1-Dichloroethene	96	61, 63
1,1-Dichloropropene	75	110, 77
1,2,3-Trichlorobenzene	180	182, 145
1,2,3-Trichloropropane	110	75, 77
1,2,3-Trimethylbenzene	0	105
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-chloropropane (DBCP)	157	155, 75
1,2-Dichlorobenzene	146	111.148
1,2-Dichloroethane	62	98
1,2-Dichloropropane	63	112
1,3,5-Trichlorobenzene	180	145, 182
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	111, 148
1,3-Dichloropropane	76	78
1,4-Dichlorobenzene	146	111, 148
1,4-Dioxane	88	58, 43, 57
2,2-Dichloropropane	77	97
2-Butanone (MEK)	72	43
2-Chloro-1,3-butadiene (Chloroprene)	53	88, 90, 51
2-Chloroethyl vinyl ether	63	65, 106
2-Chlorotoluene	91	126
2-Hexanone	58	43, 57, 100
2-Methyl-2-propanol (t-butyl alcohol)	59	41, 43
2-Methylnaphthalene	142	141, 115
2-Nitropropane	43	41, 39
3,3-Dimethyl-1-butanol	57	69, 41
3-Chloro-1-propene (Allyl chloride)	76	78

4-Chlorotoluene	91	126
4-Isopropyltoluene (p-Isopropyltoluene)	119	134, 91
4-Methyl-2-pentanone (MIBK)	58	43, 100, 85
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55
Acrylonitrile	53	52, 51
Benzene	78	--
Benzyl chloride	91	126, 65, 128
Bromobenzene	77	156, 158
Bromoform	173	175, 254
Bromomethane	96	94
Butadiene	0	54
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chlorobromomethane	130	49, 128
Chlorodibromomethane	127	129
Chloroethane	64	66 (51*)
Chloroform	83	85
Chloromethane	50	52 (51*)
cis-1,2-Dichloroethene	61	96, 98
cis-1,3-Dichloropropene	75	77, 39
Cyclohexane	56	84, 41, 69
Cyclohexanone	55	42, 98
Dibromomethane	93	95, 174
Dichlorobromomethane	83	85, 127
Dichlorodifluoromethane	85	87
Dichlorofluoromethane	67	69
Ethanol	45	46
Ethyl acetate	43	45, 61, 88
Ethyl acrylate	0	55
Ethyl ether (Diethyl ether)	59	45, 74
Ethyl methacrylate	69	41, 99, 86
Ethylbenzene	91	106
Ethylene dibromide (EDB, 1,2-Dibromoethane)	107	109, 188
Hexachlorobutadiene	225	223, 227
Hexane	57	41, 43, 56
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropyl alcohol	45	59
Isopropylbenzene	105	120
Isopropylether (IPE, Diisopropyl ether))	45	87, 59
m, p-Xylene	91	106
Methacrylonitrile	41	67, 39, 52
Methyl acetate	43	74, 59
Methyl methacrylate	41	69, 100, 39
Methylcyclohexane	83	55, 98, 41
Methylene chloride	84	86, 49
Methyl-t-butyl ether (MTBE)	73	57, 43
Naphthalene	128	-
n-Butanol (n-Butyl alcohol)	56	41, 43

n-Butyl acetate	43	56, 73, 61
n-Butylbenzene	91	92, 134
n-Propylbenzene	91	120
o-Xylene	91	106
Pentachloroethane	167	165, 169, 117, 83
Propionitrile (ethyl cyanide)	54	52, 55
sec-Butylbenzene	105	134
Styrene	104	78
tert-Amyl alcohol	59	55, 73, 43
tert-Amyl ethyl ether (TAME)	73	55, 87, 43
tert-Butyl ethyl ether (ETBE)	59	87, 41
tert-Butyl formate (TBF)	59	57, 41
tert-Butylbenzene	119	91, 134
Tetrachloroethene	166	129, 131, 164
Tetrahydrofuran	42	41, 71, 72
Toluene	91	92
trans-1,2-Dichloroethene	61	96, 98
trans-1,3-Dichloropropene	75	77, 39
trans-1,4-Dichloro-2-butene	53	88, 89
Trichloroethene	130	97, 95, 132
Trichlorofluoromethane	101	103, 105
Vinyl acetate	43	86
Vinyl chloride	62	84
Internal Standards/Surrogates:		
Fluorobenzene	96	70
Chlorobenzene-d_5	117	82
1,4-Dichlorobenzene-d_4	152	115, 78
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	111	113
1,2-Dichloroethane-d_4	65	67, 51
Toluene-d_8	98	100

Attachment 2, Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Fluorobenzene	**Chlorobenzene-d_5**	**1,4-Dichlorobenzene-d_4**
1,1,1-Trichloroethane	1,1,1,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloro-1,2,2-trifluoroethane	1,1,2-Trichloroethane	1,2,3-Trichlorobenzene
1,1-Dichloroethane	1,3-Dichloropropane	1,2,3-Trichloropropane
1,1-Dichloroethene	2-Chloroethylvinylether	1,2,3-Trimethylbenzene
1,1-Dichloropropene	2-Hexanone	1,2,4-Trichlorobenzene
1,2-Dichloroethane	3,3-Dimethyl-1-butanol	1,2,4-Trimethylbenzene
1,2-Dichloroethane-d_4 (s)	4-Methyl-2-pentanone (MIBK)	1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dichloropropane	Benzyl chloride	1,2-Dichlorobenzene
1,4-Dioxane	Bromoform	1,3,5-Trichlorobenzene
2,2-Dichloropropane	Chlorobenzene	1,3,5-Trimethylbenzene
2-Butanone	Chlorodibromomethane	1,3-Dichlorobenzene

Fluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
2-Chloro-1,3-butadiene (Chloroprene)	cis-1,3-Dichloropropene	1,4-Dichlorobenzene
2-Methyl-2-propanol (tert-Butyl alcohol)	Ethyl methacrylate	2-Chlorotoluene
2-Nitropropane	Ethylbenzene	2-Methylnaphthalene
3-Chloro-1-propene (Allyl chloride)	Ethylene dibromide (1,2-Dibromoethane)	4-Chlorotoluene
Acetone	Isopropylbenzene	4-Isopropyltoluene (p-Isopropyltoluene)
Acetonitrile	m,p-Xylene	Bromobenzene
Acrolein	o-Xylene	Bromofluorobenzene (s)
Acrylonitrile	Styrene	Hexachlorobutadiene
Benzene	Tetrachoroethene	Naphthalene
Butadiene	Toluene	n-Butylbenzene
Chlorobromomethane	Toluene-d8 (s)	n-Propylbenzene
Bromomethane	trans-1,3-Dichloropropene	Pentachloroethane
Carbon disulfide		sec-Butylbenzene
Carbon tetrachloride		tert-Buylbenzene
Chloroethane		trans-1,4-Dichloro-2-butene
Chloroform		
Chloromethane		
cis-1,2-Dichloroethene		
Cyclohexane		
Cyclohexanone		
Dibromofluoromethane (s)		
Dibromomethane		
Dichlorobromomethane		
Dichlorodifluoromethane		
Dichlorofluoromethane		
Isopropyl ether (IPE, Diisopropyl ether)		
Ethanol		
Ethyl acetate		
Ethyl acrylate		
Ethyl ether (Diethyl ether)		
Hexane		
Iodomethane		
Isobutyl alcohol (Isobutanol)		
Isopropyl alcohol (Isopropanol)		
Methacrylonitrile		
Methyl acetate		
Methyl cyclohexane		
Methyl methacrylate		
Methylene chloride		
Methyl-tert-butyl ether (MTBE)		
n-Butanol (n-Butyl alcohol)		
n-Butyl acetate		
n-Heptane		
Propionitrile		

Fluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
t-Amyl alcohol		
tert-Amyl methyl ether (TAME)		
tert-Butyl ethyl ether (ETBE)		
tert-Butyl formate		
Tetrahydrofuran		
trans-1,2-Dichloroethene		
Trichloroethene		
Trichlorofluoromethane		
Vinyl acetate		
Vinyl chloride		

18.0 Revision History

- Revision 12, 10 October 2008
  - Integration for TestAmerica and STL operations.
  - Insert corrective action procedures
- Revision 13, 25 September 2009
  - Move QC summary table and QC sample preparation instructions into Section 9.
  - Addition of new analytes: 3,3-Dimethyl-1-butanol (SC) and 1,3,5-Trichlorobenzene (NH).
  - Addition of Attachment 3 for South Carolina.
- Revision 14, 6 November 2009
  - Corporate review.
  - Addition of single-point surrogate calibration.
  - Incorporate Michigan GRO requirements.
- Revision 15, 30 October 2010
  - Addition of Amendments a (SC PQLs, Attachment 3), b (characteristic ions for 1,2,3-Trichloropropane), and c (WI soil extraction procedure).
  - Addition of new analytes: 1-Methylnaphthalene (New Mexico), Pentane, Octane, Nonane (Paraffin group).
  - Addition of QAF-45 and Section 14.2.
- Revision 16, 30 September 2011
  - Organizational changes.
  - Addition of requirement for non-preserved sample if 2-Chloroethyl vinyl ether is analyzed.
  - Addition of Minnesota Ethanol analysis requirements to Section 16.0.
  - Addition of reference to SOPs Calibration Curves (General) and Acceptable Manual Integration Practices / CA-Q-S-002.
  - Addition of 2-Chloroethyl vinyl ether, Acrolein, and Acrylonitrile preservation information.
- Revision 17, 29 February 2012
  - Organizational changes.
  - Addition of Bromoform Breakdown Check.
  - Remove paraffin standard reference.
  - TPH-GRO: change CCC frequency.
  - Corrected weighting equations.
- Revision 18, dated 30 August 2013
  - Organizational changes.
  - Specify that $r^2 \geq 0.990$.
  - OK no longer limits batch size to 10 samples.
  - Add Amendment a.

- Add 8260C.
- Add new standards and new analytes.
- Change the LCS, MS, MSD to use the primary standard.
- MA also requires LCSD.
- Addition of GRO standards

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **6010 / NV06-44.14a** Effective Date: **6/28/2013**

Last Mod. Date: 5/31/13

SOP Title**: Method 6010B/C: Inductively Coupled Plasma-Atomic Emission Spectrometry**

Affected SOP Section Number(s): Sections 6.2, 10.1

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 06

Revision Number with Mod ID: **14b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change. Add underlined text; delete crossed-out text.

Section 6.2, Supplies

- Syringe filter, PTFE membrane.

Section 10.1, Sample Preparation, add the following in a paragraph below the sample size table:

Allow any undissolved material to settle overnight, filter using a PTFE membrane, or centrifuge a portion of the prepared sample until clear. Record the filter ID and lot number The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted sample cannot be characterized, all analyses should be performed as soon as possible after the completed preparation. If any sample is filtered, the Method Blank and LCS must also be filtered.

	6/24/13		6/21/13
Supervisor Approval	Date	Technical Manager Approval Quality Assurance Approval	Date

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **6010 / NV06-44.14** Effective Date: **5/31/2013**

Last Mod. Date: 9/14/12

SOP Title**: Method 6010B/C: Inductively Coupled Plasma-Atomic Emission Spectrometry**

Affected SOP Section Number(s): Sections 1.1, 6.1, 9.2, 10.3, 15.3

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 06

Revision Number with Mod ID: **14a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change. Add underlined text; delete crossed-out text.

- Section 1.1, Analyte, Matrices, Table 1: Remove references to ICP2.
- Section 6.1, Instrumentation, Table 3, Remove ICP2 column.
- Section 9.2, Instrument QC, Linear Range bullet, Lower Limit of Quantitation Check Sample (LLQC) bullet:
  - **Linear Range Standard (LRS):** ~~For single-point calibration, run at the beginning of the analytical sequence.~~ ~~Run~~ Use the highest standard level, to show linearity to that concentration.
  - **Lower Limit of Quantitation Check Sample (LLQC):** The lower limit of quantitation check (LLQC) sample is analyzed once daily. This check sample and the low-level calibration verification standard are prepared at the same concentrations ~~(Table 8)~~ with the only difference being the LLQC sample is carried through the digestion procedure.
- Section 10.3, Calibration Table, edit steps 1 and 5:

1	See Table 5 ~~8~~ for calibration standard preparation.
5	Run LRS standards and evaluate ~~by the criteria in Section 9~~.

- Section 15.3, ~~**TestAmerica Nashville's Control Limits Manual.**~~

5/22/13		5/22/13	
Supervisor Approval	Date	Technical Manager Approval Quality Assurance Approval	Date

Title: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY SW-846 METHOD 6010B/C

Approvals (Signature/Date)	
Rodney Street Department Supervisor	8-3-12 Date
Johnny Davis Health & Safety Manager / Coordinator	8-3-12 Date
Michael H. Dunn Quality Assurance Manager	9-7-12 Date
Michael H. Dunn Technical Director	8-3-12 Date

1.0 Scope and Application

1.1 Analyte, Matrices: The method is applicable to the elements listed below. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and SPLP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been pre-filtered and acidified do not need acid digestion. Refer to SOPs Method 3010 / NV06-18, 3015 / NV06-19, Method 3050 / NV06-93, 3051 / NV06-94, and 3005 / NV06-103 for the appropriate digestion procedures.

Table 1: Recommended Wavelengths and Typical Reporting Limits

Element	CAS #	Wavelength[a] (nm)	Typical RL (µg/L)	Typical RL (mg/kg)
Aluminum	7429-90-5	308.215	100	20
Antimony	7440-36-0	206.833	10	10
Arsenic	7440-38-2	189.0	10	1.0
Barium	7440-39-3	493.4 ICP2 233.5 ICP4,5	10	2.0
Beryllium	7440-41-7	313.042	4	1.0
Boron	7440-42-8	249.678x2	50	10
Cadmium	7440-43-9	226.502	1	1.0
Calcium	7440-70-2	373.6 ICP4, 5 317.933	1000	100
Chromium	7440-47-3	267.716	5	1.0
Cobalt	7440-48-4	228.616	20	3.0
Copper	7440-50-8	324.754	10	2.0
Iron	7439-89-6	271.4 ICP2,4,5 259.9 ICP4,5	100	20
Lead	7439-92-1	220.353	5	1.0
Lithium	7439-93-2	670.784	50	10
Magnesium	7439-95-4	279.079	1000	100
Manganese	7439-96-5	257.610	15	3.0
Molybdenum	7439-98-7	202.030	50	3.0
Nickel	7440-02-0	231.604x2	10	1.0
Potassium	7440-09-7	766.491	1000	100
Selenium	7782-49-2	196.026	10	2.0
Silver	7440-22-4	328.068	5	1.0
Sodium	7440-23-5	589.5[b] ICP ICP4,5 330.2 ICP2,4,5.	1000	200
Strontium	7440-24-6	421.5	50	10
Sulfur	7704-34-9	182.0	500	2.0
Thallium	7440-28-0	190.864	10	10
Tin	7440-31-5	189.980x2	50	10
Titanium	7440-32-6	334.941	50	10
Vanadium	7440-62-2	292.402	20	10
Zinc	7440-66-6	213.856x2 ICP4,5 206.2 ICP2	50	IS
Yttrium	7440-65-5	224.3 ICP4,5 360.0 ICP4,5	IS	IS
Scandium	7440-20-2	361.384 ICP2	IS	IS
Indium	7440-74-6	230.6	IS	

[a]The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e. g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. In time, other elements may be added as more information becomes available and as required.
[b]Highly dependent on operating conditions and plasma position.

1.2 Reporting Limits: Detection limits and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the analytical wavelengths and typical reporting limits in clean matrices.

1.3 Use of this method is restricted to analysts who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 Prior to analysis, samples are solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This method describes multi-elemental determinations by ICP-AES using simultaneous and sequential systems. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, is determined by the complexity of the spectrum adjacent to the analyte line. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

3.0 Definitions

3.1 Field Reagent Blank: An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose is to determine if method analytes or other interferences are present in the field environment.

3.2 Instrument Detection Limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit.

3.3 Internal Standard: Pure analyte added to a sample, in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

3.4 Linear Range (LR): The concentration range over which the instrument response to an analyte is linear.

3.5 Spectral Interference Check (SIC) Solution: Used to prepare ICSA and ICSAB. A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known inter-element spectral interferences with respect to a defined set of method criteria. See Thermo Jarrell Ash method 136972-00 revision 0 (7/28/95) for

procedure.

3.6 Inter-element Correction (IEC): Single element solutions are used to determine the appropriate location for background correction and to establish the inter-element correction routine.

3.7 Toxicity Characteristics Leaching Procedure (TCLP): An extraction process which attempts to simulate the leaching of samples into the ground/soil at a municipal landfill.

3.8 TCLP Blank Matrix: An aliquot of TCLP fluid that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. This blank is used to determine if method analytes or other interferences are present in the TCLP extraction fluid used in the preparation of TCLP samples.

3.9 Synthetic Precipitation Leaching Procedure (SPLP): An extraction process which attempts to simulate the leaching of samples in to the soil from a rain event.

3.10 SPLP Blank Matrix: An aliquot of SPLP fluid that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. This blank is used to determine if method analytes or other interferences are present in the SPLP extraction fluid used in the preparation of SPLP samples.

3.11 See TestAmerica Nashville's QA Manual Appendix 5 for other laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. The locations selected for the measurement of background intensity are determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

4.1.2 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences produce false positive determinations and are reported as analyte concentrations. Analysts may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. The interferences listed are only those that occur between method analyses. Only interferences of a direct overlap nature are listed.

Table 2: Potential Interferences by Analyte

Analyte	Wavelength (ηm)	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	XX	--	--	XX
Antimony	206.833	XX	--	XX	--	XX	--	--	--	XX	XX
Arsenic	189.0	XX	--	XX	--	--	--	--	--	XX	
Barium	493.4	--	--	--	--	--	--	--	--	--	--

Analyte	Wavelength (ηm)	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Beryllium	313.042	--	--	--	--	--	--	--	--	XX	XX
Cadmium	226.502	--	--	--	--	XX	--	--	XX	--	--
Calcium	317.933	--	--	XX	--	XX	XX	XX	--	XX	XX
Chromium	267.716	--	--	--	--	XX	--	XX	--	--	XX
Cobalt	228.616	--	--	XX	--	XX	--	--	XX	XX	--
Copper	324.754	--	--	--	--	XX	--	--	--	XX	XX
Iron	271.4	--	--	--	--	--	--	XX	--	--	--
Lead	220.353	XX	--	--	--	--	--	--	--	--	--
Lithium	670.784										
Magnesium	279.079	--	XX	XX	--	XX	--	XX	--	XX	XX
Manganese	257.610	XX	--	XX	--	XX	XX	--	--	--	--
Molybdenum	202.030	XX	--	--	--	XX	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	XX	--	--	--	XX	--	--	--	--	--
Sodium	588.995 / 330.2	--	--	--	--	--	--	--	--	XX	--
Sulfur	182.034										
Thallium	190.864	XX	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	XX	--	XX	--	--	--	XX	--
Zinc	213.856 / 206.2	--	--	--	XX	--	--	--	XX	--	--
Dashes indicate that no interference was observed even when interferants were introduced.											
XX - Interference may be possible.											

4.1.3 When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i. e., false analyte concentrations) arising from the linear range of the interference element. Instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects **must** be evaluated for each individual instrument since the intensities vary. This evaluation is filed in the method of the instrument's computer.

4.1.4 Inter-element correction accuracy must be verified daily by analyzing spectral interference check solutions. Inter-element correction factors must be verified every **six** months or when ICSA or ICSAB fail(s) criteria.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Physical interferences are reduced by diluting the sample and by using an internal standard.

4.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by matrix matching. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory interferences result when analyses in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. This method requires a rinse period of at least 45 seconds between samples and standards.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements:

- The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- The inductively coupled plasma should only be viewed with proper eye protection from the ultraviolet emissions.
- The ICP uses high voltage.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors causes coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Causes redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors causes breathing difficulties and leads to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Causes redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Inductively coupled plasma emission spectrometer, Thermo Scientific ICAP 6000 Series, or equivalent.
  - Computer-controlled emission spectrometer with background- correction capability. The spectrometer must be capable of meeting and complying with the requirements described and referenced in Section 4.0.
  - Radio-frequency generator compliant with FCC regulations.
  - Argon gas supply - High purity grade (99.99%).
  - A peristaltic pump is required to deliver both internal standard and sample solutions to the nebulizer.
  - Mass flow controller to regulate the argon flow rate, especially the aerosol transport gas.

- See Table 3 for instrument operating conditions selected as being optimal to provide the lowest reliable instrument detection limits and method detection limits.

Table 3: Inductively Coupled Plasma Instrument Operating Conditions

	ICP2	ICP4 & ICP5
Incident rf power	1100 watts	1150 watts
Reflected rf power	< 5 watts	--
Viewing height above work coil	15 mm	--
Injector tube orifice i.d.	1 mm	2 mm center tube
Argon supply	liquid Argon	liquid Argon
Argon pressure	40 psi	80+ psi
Coolant argon flow rate	19 L/min	12 L/min
Aerosol carrier argon flow rate	620 mL/min	0.5 L/min
Auxiliary (plasma) argon flow rate	300 mL/min	0.5 L/min
Sample uptake rate controlled to	1.2 mL/min	50 rpm

- Specific wavelengths are listed in Table 1.
- **Optimization of the plasma operating conditions:** This function may be performed at any time and is a routine which ensures that the wavelengths are correctly located on the detector. During this routine, the pump stops, and the nebulizer gas is turned off because the routine uses plasma wavelength positions, so no sample is required. The routine runs automatically when the plasma is ignited.

- Analytical balance, with capability to measure to 1 mg.

6.2 Supplies

For determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) must be sufficiently clean for the task objectives.

- Volumetric flasks, 25 mL, 100 mL, 200 mL, Class A.
- Adjustable Eppendorf pipettors, 10 µL - 100 µL, 100 µL - 1000 µL, with disposable plastic tips.
- Disposable serological pipettes, 1 mL, 5 mL, 10 mL.
- Graduated Cylinders, 50 mL, 250 mL, 500 mL, Class A.
- Beakers, 150 mL, with ribbed watch glass.
- Centrifuge tubes, plastic, 50 mL, certified, graduated, with screw caps.
- Watch glass, plastic, ribbed (for use with the centrifuge tubes).
- Plastic centrifuge tube racks.
- Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125 L to 1-L capacities.
- One-piece stem FEP wash bottle with screw closure, 125 L capacity.
- pH test strips.
- Teflon™ boiling chips for solid matrix blank (Chemware P/N D1069103, or equivalent).

7.0 Reagents and Standards

7.1 Reagent water, analyte-free.

7.2 Reagents may contain elemental impurities, which might affect analytical data. Only high-purity reagents that conform to the American Chemical Society specifications are used. If the purity of a reagent is in question, analyze for contamination. All acids used for this method **must be** of ultra high-purity grade or equivalent.

7.3 **Hydrochloric acid**, concentrated (sp.gr. 1.19), HCl. To prepare a 5% solution, add 10 mL concentrated HCl to 190 mL reagent water.

7.4 **Nitric acid**, concentrated (sp.gr. 1.41), HNO_3. To prepare a 5% solution, add 10 mL concentrated HNO_3 to 190 mL reagent water.

7.5 **Standard Stock Solutions**: Stock standards are purchased as certified commercial solutions at 100, 500 or 1000 µg/mL (recommended). Stock solutions are stored in FEP bottles. Replace stock standards yearly when succeeding dilutions for preparation of calibration standards cannot be verified. Store standards containing silver in the dark.

7.6 **Spectral Interference Check solutions** are prepared in the same acid mixture as the calibration standards and stored in FEP bottles. See Table 6 for SIC Preparation (ICSA and ICSAB).

7.7 **Internal Standards**: Yttrium and Indium for ICP4. Purchase Ultra Scientific, or equivalent, commercial standards at 1000 µg/mL in 2% HNO_3: IAA-249-5 for Indium; IAA-239-5 for Yttrium. Dilute 5 mL to 1 liter with 5% HNO_3 for ICP2. For ICP4 and ICP5, dilute 5 mL Yttrium and 30 mL Indium to 1 liter with 5% HNO_3.

7.8 **Inter-element Correction (IEC) Single-element Standards**: Purchase the following single-element standards at 1000 µg/mL from Ultra Scientific, or equivalent:

Element	Catalog #	Element	Catalog #	Element	Catalog #
Aluminum	IAA-213	Copper	IAA-229	Silicon	IAA-214
Antimony	IAA-251	Iron	IAA-226	Silver	IAA-247
Arsenic	IAA-233	Lead	IAA-282	Sodium	IAA-211
Barium	IAA-256	Lithium	IAA-203	Strontium	IAA-238
Beryllium	IAA-204	Magnesium	IAA-212	Sulfur	IAA-016
Boron	IAA-205	Manganese	IAA-225	Thallium	IAA-281
Cadmium	IAA-248	Molybdenum	IAA-242	Tin	IAA-250
Calcium	IAA-220	Nickel	IAA-228	Titanium	IAA-222
Chromium	IAA-224	Potassium	IAA-219	Vanadium	IAA-223
Cobalt	IAA-227	Selenium	IAA-234	Zinc	IAA-230
				Zirconium	ICP-040

7.9 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water, TCLP Extract	HDPE or Glass	125 mL	HNO_3 to pH ≤ 2[1]	6 months	SW-846 Chapter 2
Soil	HDPE or Glass	50 grams	No requirement	6 months	SW-846 Chapter 2

[1]If water samples are preserved in the lab, they should be held for at least 24 hours before analysis; record acidification start/stop time and pH. Temperature preservation is not required.

9.0 Quality Control

Refer to the quality control section of TestAmerica-Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples must be prepared with each batch of no more than 20 samples:			
Quality Controls	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Method Blank	1 each batch	< ½ RL or MDL whichever is greater	Re-analyze. If contamination persists, correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. If target > 10X blank, acceptable to report.
Laboratory Control Sample (LCS)[1], second source	1 each batch	80-120%[3] recovery	Correct problem then re-prep and analyze the LCS and all affected targets in the affected analytical batch. If high and ND, OK to report. For 6010C, LCS may be re-analyzed once.
Matrix Spike	1 each batch	75-125%[3] recovery	Perform post-digestion spike.
Matrix Spike Duplicate	1 each batch	75-125%[3] recovery <20[2]% RPD	Perform post-digestion spike.
Post digestion spike addition	When MS/MSD fail.	Recovery within 25% for 6010B and within 20% for 6010C of the expected results.	Perform dilution test.
Dilution test	If MS/MSD fail.	1:4 fold dilution (5X) must agree within 10% of the original determination.	Qualify results.

[1]AZ, TX, WV require an LCS duplicate in each batch.
[3]If historical limits are calculated, they cannot exceed these limits.

- **Method Blank:** The laboratory prepares and analyzes one blank (reagent water or Teflon™ boiling chips) with each batch of up to 20 samples of the same matrix.
- A **Laboratory Control Sample (LCS)** must be analyzed with every batch. See Table 4 for LCS preparation using 50 mL reagent water for water batches and 0.5 gram Teflon™ chips for soil batches.
- **Matrix Spike / Matrix Spike Duplicate:** Analyze a matrix spike and matrix spike duplicate at a frequency of one per matrix batch up to 20 samples. In each case the MS aliquot must be a duplicate of the aliquot used for sample analysis and for total recoverable determinations added prior to sample preparation. See SOP Sample Homogenization, Subsampling, and Compositing / NV08-229. The added analyte concentration and standard source must be the same as that used in the LCS (Table 4).
  - For each batch, check for matrix effects as follows:
    - If MS/MSD is outside the QC limits, the same sample from which the MS/MSD aliquots were prepared is also spiked with a **post-digestion spike** (i. e., nominally add 250 µL of the LCS/MS/MSD solution to 25 mL digested aliquot). Otherwise, another sample from the same preparation is used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and is recovered as described in the above table. The spike addition produces a minimum level of 10 times and a

maximum of 100 times the lower limit of quantitation. If this spike fails, then the dilution test is run on this sample. If both the MS/MSD and the post-digestion spike fail, then matrix effects are confirmed.

- **Dilution test**: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (5X) should agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.

9.2 Instrument QC

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Inter-element Correction, single element standards	6 months	± RL	Alter wavelength or background correction.
Spectral Interference Check Solutions, A and AB	Beginning and end of each day or every 8 hours	Target ± 2 times RL or ± 20% true.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples.
Instrument Detection Limits (IDL)	Quarterly	±3 standard deviations of the average response.	Re-run IDL. If > MDL, adjust MDL to equal IDL.
Independent Calibration Verification Sample (ICV), second source	Immediately after calibration	90-110 % recovery	Correct problem then repeat initial calibration.
Independent Calibration Blank (ICB)	Immediately after ICV	No target analytes above lower limit of quantitation	Correct problem, re-calibrate.
Linear Range Standard	Daily	90-110%	Repeat calibration (ICP2).
Continuing Calibration Verification Sample (CCV)	Every 10 samples and at the end of the run	90-110% true	Re-analyze once. If fails again, then repeat calibration and re-analyze all samples since last successful CCV.
Undigested Low Level Continuing Calibration Verification (LLCCV)	Beginning and end of each batch.	70-130% true	Re-calibrate.
Continuing Calibration Blank	Following the CCV	No target analytes above lower limit of quantitation	Correct problem then analyze calibration blank and previous 10 samples.
MDL Verification (digested)	Yearly	Detected	Re-evaluate MDL standard used and MDL; see Technical Director
Digested Lower Limit of Quantitation Check (LLQC) or Report Limit Verification (RLV)	Once daily	70-130% true	Re-calibrate.
Internal Standards	All samples, standards, QC	60-140% true	Dilute and re-run. For blank and LCS, correct problem and re-run batch.

- **Inter-element Correction (IEC):** When correction is appropriate, the concentration of all targets must be within ± the RL, i. e., RL = 0.01, acceptance is -0.01 to +0.01. Once established, the entire routine must be initially and periodically verified every six months, or whenever there is a change in instrument operating conditions or when ICSA or ICSAB fail criteria.

Single element standard concentrations that must be analyzed every six months are shown as follows:

Element	Required Final Concentration (mg/L)	Stock Standard Solution (µg/mL)	Volume of Stock Standard Solution (mL)	Final Volume (mL)
Aluminum	50	1000	25	50
Antimony	10	1000	0.5	50
Arsenic	50	1000	2.5	50
Barium	50	1000	2.5	50
Beryllium	10	1000	0.5	50
Boron	50	1000	2.5	50
Cadmium	10	1000	0.5	50
Calcium	100	1000	5.0	50
Chromium	50	1000	2.5	50
Cobalt	50	1000	2.5	50
Copper	50	1000	2.5	50
Iron	100	1000	5.0	50
Lead	50	1000	2.5	50
Lithium	25	1000	1.25	50
Magnesium	100	1000	5.0	50
Manganese	10	1000	0.5	50
Molybdenum	10	1000	0.5	50
Nickel	50	1000	2.5	50
Potassium	100	1000	5.0	50
Selenium	20	1000	1.0	50
Silicon	50	1000	2.5	50
Silver	10	1000	0.5	50
Sodium	100	1000	5.0	50
Strontium	50	1000	2.5	50
Sulfur	100	1000	5	50
Thallium	20	1000	1.0	50
Tin	50	1000	2.5	50
Titanium	50	1000	2.5	50
Vanadium	50	1000	2.5	50
Zinc	10	1000	0.5	50
Zirconium	50	1000	2.5	50

If interferences are observed, they must be mitigated by the use of interference correction equations or by changing to a different wavelength. The objective is to reduce interferences.

Required ICSA and ICSB solutions are described in Table 6 for daily verification. Initial and periodic verification data of the routine are filed in the maintenance of the instrument's computer.

- **Spectral interference check solution (ICSA and ICSAB):** The laboratory must periodically verify the inter-element correction routine by analyzing SIC solutions. The spectral interference check solution is run at the beginning and end of the day's sequence or every 8 hours, whichever is more frequent. If the SIC does not meet criteria, then the SICs are re-

analyzed. These solutions are used to periodically verify a partial list of the on-line (and possible off-line) inter-element spectral correction factors for the recommended wavelengths given in Table 1.

NOTE: The SIC solution must be analyzed more than once to confirm a change has occurred with adequate rinse time between solutions and before subsequent analysis of the calibration blank.

- Ensure that the analytical results of ICS Solution A (ICSA) fall within the control limit of ± 2 times the RL of the analyte's true value or ± 20% of the analyte's true value, whichever is greater (the true value is zero unless otherwise stated) in the ICSA. For example, if the analysis result(s) for Arsenic (RL = 10 µg/L, ICSA true value = 0 µg/L) in the ICSA analysis during the run is 19 µg/L, then the analytical result for Arsenic falls within the ± 2 times the RL window for Arsenic in the ICSA. If the analytical results of the ICSA do not fall within the control limits, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze the analytical samples analyzed since the last compliant ICSA was performed.
- Ensure that the results for the ICS Solution AB (ICSAB) during the analytical runs fall within the control limit of ± 2 times the RL of the true value or ± 20% of the true value, whichever is greater, for the analytes included in the ICSAB. If the analytical results of the ICSAB do not fall within the control limits, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze the analytical samples analyzed since the last compliant ICSAB was performed.

- **Instrument Detection Limits (IDL):** IDLs in µg/L are estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Perform each measurement as though it were a separate analytical sample (i. e., follow each measurement by a rinse and/or any other procedure normally performed between the analysis of separate samples). Determine IDLs at least every three months and keep with the instrument logbook. Compare the calculated IDLs to the MDLs. MDLs are equal to or greater than the IDL.
- **Independent Calibration Verification (ICV and ICB):** The laboratory analyzes the ICV and a calibration blank (ICB) immediately following daily calibration. See Table 7 for ICV preparation using a second-source standard. The ICB must not contain target analytes above the lower limit of quantitation.
- **Linear Range Standard (LRS):** For single-point calibration, run at the beginning of the analytical sequence. Run the highest standard level, to show linearity to that concentration. All samples exceeding **90%** this concentration are diluted. The LRS concentrations are Al, Fe, Ca and Mg at 200 µg/mL, Na, K, at 100 µg/mL, Ba at 50 µg/mL, Ag at 5 µg/mL, and all other elements at 10 µg/mL. The standard must be within 10% of the true values to continue.
- **Undigested Low-Level Continuing Calibration Verification (LLCCV):** Analyze an undigested LLCCV at the beginning and end of each batch. Prepare it from the primary calibration standard at the RL.
- **Continuing Calibration Verification (CCV and CCB):** Analyze after every 10th sample and at the end of the analytical sequence.
  - See Table 5 for CCV preparation using the primary source standard at the mid-point of the calibration curve. All samples **must be bracketed** by acceptable CCVs and CCBs.
  - The CCB (prepared by adding 25 mL concentrated HNO_3 to 500 mL reagent water) must not contain target analytes above the MDL or ½ the RL, whichever is greater. If the criterion is not met, terminate the analysis, correct the problem, and re-analyze the previous 10 samples.

- **MDL Verification:** A solution containing all target analytes at 2-3 times the MDL must be **digested** and analyzed after the completion of the MDL study and on an annual basis. Detection limits are verified when all analytes in the MDL check solution are detected.
- **Lower Limit of Quantitation Check Sample (LLQC):** The lower limit of quantitation check (LLQC) sample is analyzed once daily. This check sample and the low-level calibration verification standard are prepared at the same concentrations (Table 8) with the only difference being the LLQC sample is carried through the digestion procedure.
- **Internal Standards:** Use the internal standard technique by adding one or more elements (not in the samples and verified not to cause an uncorrected inter-element spectral interference) at the same concentration (which is sufficient for optimum precision) to the prepared samples (blanks and standards) that are affected the same as the analytes by the sample matrix. Use the ratio of analyte signal to the internal standard signal for calibration and quantitation. Internal standards (Yttrium and Indium) are automatically added to all calibration standards, samples, and QC, by the instrument.

10.0 Procedure

10.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been pre-filtered and acidified do not need acid digestion. Samples which are not digested must use an internal standard. See Section 1.1 for digestion SOPs.

Matrix	Sample Size
Water	50 mL of sample
TCLP Extract	10 mL of extract
Soil	0.5 gram of sample

10.2 Instrument Setup

1	Inspect the sample introduction system including the nebulizer, torch, center tube, and uptake tubing for salt deposits, dirt and debris that would restrict solution flow and affect instrument performance. Clean the system when needed or on a daily basis.
2	Power up all accessories and the unit. Allow the instrument to become thermally stable before beginning (usually requiring about 30 minutes of operation prior to calibration).
3	Click on the plasma icon in the lower right of the Analyst screen. Then click the instrument status button. Make sure all interlocks have a green light. Then push the plasma on button. Confirm flow to the plasma.
4	To shutdown, click the plasma icon and push the plasma off button.
5	Instrument is automatically optimized when the program is opened.
6	Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The analyst must follow the instructions provided by the instrument manufacturer and this SOP. For the 6500 series, the conditions usually vary from 750-1350 watts power, 10-20 liters/minute coolant gas flow, 0.25-1.5 liters/minute nebulizer gas flow, 0.25-2 liters/minute auxiliary gas flow, 20-125 rpm pump rate with a 40 second flush time and 30 seconds maximum integration time. Perform two replicate integrations; report the average.
7	Establish sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

8	All samples that exceed the upper calibration standard must be diluted and re-analyzed or a linear range standard must be run with 10% of the true value.

10.3 Calibration: Refer to SOPs Calibration Curves (General) / CA-Q-S-005 and Selection of Calibration Points / CA-T-P-002. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1	See Table 8 for calibration standard preparation. For multi-point calibration, use first-order linear regression (**$r \geq 0.998$, $r^2 \geq 0.996$**). The lowest non-zero standard concentration is considered the lower limit of quantitation, i. e., RL. Higher order fits are not allowed.
2	The absolute value of the results of the calibration blank is less than the value of the MDL.
3	After initial calibration, the calibration curve must be verified by use of an initial calibration verification (ICV) standard. The calibration curve must be verified at the end of each analysis batch and after every 10 samples by use of a continuing calibration verification (CCV) standard and a continuing calibration blank (CCB).
4	The calibration curve must also be verified prior to the analysis of any samples and at the end of the batch by use of a low-level continuing calibration verification (LLCCV) standard.
5	Run LRS standards and evaluate by the criteria in Section 9.
6	Verify the inter-element correction factors at the beginning and end of the daily sequence or every 8 hours.

10.4 Sample Analysis

- Follow the analysis sequence in Table 9.
- Samples exceeding the 90% of the LRS are diluted and rerun.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{\% Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Response of the analyte.
A_{is} = Response of the internal standard.
C_s = Concentration of the analyte.
C_{is} = Concentration of the internal standard

11.4 % Drift

% Drift = (Result - True Value) x 100
True Value

11.5 **Linear Calibration Using a Least Squares Regression**: This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$y = ax + b$

y = instrument response (peak area)
a = slope of the line
x = concentration of the calibration standard
b = the intercept

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit.

11.6 **Coefficient of Determination** **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response ratio
x = Concentration

11.7 **Concentration:** Sample data are reported in units of mg/L for aqueous samples, mg/kg for solid samples. LIMS calculates the concentration from the raw data provided by the analyst.

Concentration (mg/L or mg/kg) = (µg/mL* from instrument)(digest volume, mL)(Dilution factor)
Sample Volume (mL) or Mass (g)

µg/mL from instrument = y = mx + b

***average of two replicates**

11.8 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the MDL.

11.9 Account for any additional dilution of the prepared sample solution needed to complete the determination of analytes exceeding 90% or more of the LRS upper limit. Do not report data below the determined analyte MDL concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required to complete the analysis.

12.0 **Method Performance**

12.1 **Method Detection Limit Study (MDL):** The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99%

confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

Quarterly, run 7 IDLs on three non-consecutive days; calculate the standard deviation per day. Compare three times the average of the standard deviations to the MDL. If 3xIDL > MDL, rerun the IDLs. If the finding persists, select the higher of the two as the "working MDL" for that quarter.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

12.5 Control Charts: Laboratory method performance can be shown with the use of control charts, available from LIMS or the QA department.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Acidic aqueous wastes are taken to the waste disposal area, neutralized, and discharge to the sanitary sewer.

15.0 References / Cross-References

15.1 Method 6010B, SW-846 Update III, Revision 2, December 1996, and **Method 6010C**, SW-846 Update IV, Revision 3, February 2007.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 TestAmerica Nashville's Control Limits Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Calibration Curves (General) / CA-Q-S-005, Selection of Calibration Points / CA-T-P-002, Method 3005 / NV06-103; Method 3010 / NV3010; Method 3050 / NV06-93; Method 3051

/ NV06-94; Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Determination of Method Detection Limits / NV08-202, Sample Homogenization, Subsampling, and Compositing / NV08-229, Reagent and Standard Purchase / NV08-214.

15.5 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

Item	Modification
1	If 3030C digestion is specified, see the attachment in SOP 3005 / NV06-103 for that procedure.
2	For OK, NV, SC, and WY samples, reference 6010C. For all other states, reference 6010B.
3	Corporate Quality Memorandum CA-Q-QM-004, Technical Guidance on Checking for Spectral Interferences in Optical ICP Analysis, September 24, 2009.

17.0 Attachments

Table 4: LCS and MS/MSD Spiking Solution
Environmental Express HP3948
Use 0.5 mL as received

Analyte	Stock Std Concentration (µg/mL)	Final Concentration of Digestate (µg/mL)
Aluminum	200	2.0
Antimony	10	0.1
Arsenic	5	0.05
Barium	200	2.0
Beryllium	5	0.05
Boron	100	1.0
Cadmium	5	0.05
Calcium	500	5.0
Chromium	20	0.2
Cobalt	50	0.5
Copper	25	0.25
Iron	100	1.0
Lead	5	0.05
Lithium	100	1.0
Magnesium	500	5.0
Manganese	50	0.5
Molybdenum	50	0.5
Nickel	50	0.5
Potassium	500	5.0
Silver	5	0.05
Sodium	500	5.0
Sulfur	100	1.0
Thallium	5	0.05
Titanium	100	1.0
Vanadium	50	0.5
Zinc	50	0.5

Table 5: Calibration Solutions

Analyte	ICAL 1	ICAL 2	ICAL 3	ICAL 4	ICAL 5
Al, Sb, Ar, Ba, Be, B, Cd, Co, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, NI, Ag, Tl, S, Sn, Ti, V, Zn	ICUS 2648: 2.0 µg/mL	Dilute 20 mL ICUS 2648/ 40 mL: 1.0 µg/mL	Dilute 10 mL ICUS 2648/ 40 mL: 0.5 µg/mL		
Ba	ICUS 3033: 10.0 µg/mL				
Al	ICUS 3033: 10.0 µg/mL	ICUS 2614: 500 µg/mL			
K	ICUS 3033: 10.0 µg/mL	ICUS 2614: 100 µg/mL			
Mg,	ICUS 3033: 10.0 µg/mL	ICUS 2614: 500 µg/mL			
Ca	ICUS 3033: 10.0 µg/mL		5 mL 1000 µg Ca /mL stock/50 mL: 100 µg/mL	15 mL 1000 µg Ca /mL stock/50 mL: 300 µg/mL	ICUS 2614: 500 µg/mL
Fe	ICUS 3033: 10.0 µg/mL	2.5 mL 1000 µg Fe/mL stock/50 mL: 50 µg/mL	5 mL 1000 µg Fe /mL stock/ 50 mL: 100 µg/mL	ICUS 2614: 200 µg/mL	
Na	ICUS 3033: 10.0 µg/mL		5 mL 1000 µg Na /mL stock/ 50 mL: 100 µg/mL	15 mL 1000 µg Na /mL stock/ 50 mL: 300 µg/mL	ICUS 2614: 500 µg/mL
Sulfur	0.02 mL/40 mL 0.5 µg/mL	0.1 mL/50 mL 2.0 µg/mL	ICUS 3033 10.0 µg/mL	2.5 mL/50 mL 50 µg/mL	5 mL/50 mL 100 µg/mL
Silicon	0.5 mL 1000 µg/mL stock/ 10.0 µg/mL				

CCV and RLV Sources and Concentrations

Analyte	CCV ICUS-2613 (µg/mL)	RLV ICUS-2647 (µg/mL)
Aluminum	10.0	0.1
Antimony	1.0	0.01
Arsenic	1.0	0.01
Barium	2.0	0.01
Beryllium	1.0	0.004
Boron	1.0	0.05

Analyte	CCV ICUS-2613 (µg/mL)	RLV ICUS-2647 (µg/mL)
Cadmium	1.0	0.001
Calcium	10.0	1.0
Chromium	1.0	0.005
Cobalt	1.0	0.02
Copper	1.0	0.01
Iron	10.0	0.05
Lead	1.0	0.005
Lithium	1.0	0.05
Magnesium	10.0	1.0
Manganese	1.0	0.015
Molybdenum	1.0	0.05
Nickel	1.0	0.01
Potassium	10.0	1.0
Selenium	1.0	0.01
Silver	1.0	0.005
Sodium	10.0	1.0
Strontium	1.0	0.05
Sulfur	1.0	0.5
Thallium	1.0	0.01
Tin	1.0	0.05
Titanium	1.0	0.05
Vanadium	1.0	0.02
Zinc	1.0	0.05

Table 6: Spectral Interference Check Solutions

ICSA Standard

ICSA Stock, SPEX # INT-A1

ICSA Solution: Dilute 25 mL of ICSA Stock + 12.5 mL HNO_3 to 250 mL with reagent water.

Analyte	ICSA Stock Conc. (µg/mL)	ICSA Conc. (µg/mL)
Aluminum	5000	500
Calcium	5000	500
Magnesium	5000	500
Iron	2000	200

ICSAB Standard

ICSA Stock, SPEX # INT-A1
ICSB Stock, SPEX # INT-B1
ICSB2 Stock, SPEX # INT-B2

ICSAB Solution*: Dilute 25 mL of ICSA Stock + 2.5 mL of ICSB Stock + 2.5 mL of ICSB2* Stock + 12.5 mL HNO_3 to 250 mL with reagent water.

Analyte	ICSA Stock Conc. (µg/mL)	ICSB Stock Conc. (µg/mL)	ICSB2 Stock Conc. (µg/mL)	ICSAB Conc. (µg/mL)
Aluminum	5000		100	501
Calcium	5000		10	500.1
Magnesium	5000		10	500.1
Iron	2000		10	200.1
Barium		50		0.5
Beryllium		50		0.5
Cadmium		100		1.0
Chromium		50		0.5
Cobalt		50		0.5
Copper		50		0.5
Lead		100		1.0
Manganese		50		0.5
Nickel		100		1.0
Silver		100		1.0
Vanadium		50		0.5
Zinc		100		1.0
Antimony			100	1.0
Arsenic			100	1.0
Boron			100	1.0
Molybdenum			100	1.0
Selenium			100	1.0
Silica			10	0.1
Sodium			100	1.0
Thallium			100	1.0

Table 7: ICV Standard
Source: Inorganic Ventures STLTN-CAL-3 for all except Tin
Ultra Scientific 1AA-250, 1000 µg/mL for Tin
ICV Preparation: 0.05 mL Tin standard to 50 mL STLTN-CAL-3
Environmental Express HP100054-5, 1000 µg/mL for Sulfur
ICV Preparation: 0.05 mL Sulfur standard to 50 mL STLTN-CAL-3

Analyte	STLTN-CAL-3 (µg/mL)	Ultra 1AA-250 Sn (µg/mL)	Environmental Express HP100054 (µg/mL)	ICV Conc. (µg/mL)
Aluminum	10			10
Antimony	1			1
Arsenic	1			1
Barium	2			2
Beryllium	1			1
Boron	1			1
Cadmium	1			1
Calcium	10			10
Chromium	1			1
Cobalt	1			1
Copper	1			1
Iron	10			10

Analyte	STLTN-CAL-3 (µg/mL)	Ultra 1AA-250 Sn (µg/mL)	Environmental Express HP100054 (µg/mL)	ICV Conc. (µg/mL)
Lead	1			1
Lithium	1			1
Magnesium	10			1
Manganese	1			1
Molybdenum	1			1
Nickel	1			1
Potassium	10			10
Selenium	1			1
Silver	1			1
Sodium	10			10
Strontium	1			1
Sulfur		1000		1
Thallium	1			1
Tin			1000	1
Titanium	1			1
Vanadium	1			1
Zinc	1			1

Table 8: Linear Range Standard/High Calibration Standards

Analyte	ICUS 2614 Conc. (µg/mL)
Al, Ca, Mg, Na	500
Fe	200
K	100

Table 9: Typical Analytical Sequence

	Definitions
0.0 mg/L standard(or blank)	Calibration Standard
0.5 mg/L	Calibration Standard
1.0 mg/L	Calibration Standard
2.0 mg/L	Calibration Standard
10.0 mg/L	Calibration Standard
LRS	High Range Calibration Verification
100 mg/L	Calibration Standard
300 mg/L	Calibration Standard
50 mg/L	Calibration Standard
ICV	Independent (initial) Calibration Verification
ICB	Initial Calibration Blank
RLV	Report Limit Verification
ICSA	Initial Spectral Interference Check
ICSAB	Initial Spectral Interference Check
Up to 10 samples	
MS	Matrix Spike

MSD	Matrix Spike Duplicate
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
LCS	Laboratory Control Sample
RLV (as needed)	Report Limit Verification
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
ICSA	Spectral Interference Check
ICSAB	Spectral Interference Check
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
CCV	Final Continuing Calibration Verification
CCB	Final Continuing Calibration Blank
RLV	Final Report Limit Verification
ICSA	Final Spectral Interference Check
ICSAB	Final Spectral Interference Check

18.0 Revision History

- Revision 10, dated 29 February 2008
  - Integration for TestAmerica and STL operations.
  - Addition of control limits for ICSA and ICSAB.
  - Addition of text on Spectral Interference Check Solutions
  - Change from CRDL/CRQL to CRL, current terminology
  - Addition of current catalog numbers for Table 7 individual elements.
  - Correction of order of Table 9
- Revision 11, dated 17 October 2008
  - Updated to SW-846 Update IV
- Revision 12, dated 30 October 2009
  - Inserted information on new ICV standard, new standards for ICP4.
  - Modified analytical sequence and added ICP4 sequence.
  - Reformatting and condensing.
  - Addition of requirements of Corporate Quality Memorandum CA-Q-QM-004, Technical Guidance on Checking for Spectral Interferences in Optical ICP Analysis, September 24, 2009.
  - Corporate review.
- Revision 13, dated 21 February 2011
  - Addition of QAF-45 and Section 14.2
  - Addition of Sulfur information, updated wavelengths in Table 1.
  - Addition of operating conditions optimization for ICP4 and 5.
  - New vendor IDs for elements.
  - Delete ICP1, taken out of service.
  - ICP2 new calibration standard preparation; changed SIC solution preparation.

- Revision 14, dated 14 September 2012
  - Organizational changes.
  - Amendments 13a,b,c.
  - Iron RL change from 50 to 100 µg/L; from 10 mg/kg to 20 mg/kg.
  - Removal of ICP2.
  - Addition of Sulfur throughout.
  - Remove the requirement for 10-sample batches if samples are from OK or WY.
  - Addition of SOPs Calibration Curves (General) / CA-Q-S-005, and Selection of Calibration Points / CA-T-P-002. Remove 3015 / NV06-19 (archived).

APPENDIX B

EXAMPLE CHAIN-OF-CUSTODY FORM

THE LEADER IN ENVIRONMENTAL TESTING
Nashville Division
2960 Foster Creighton
Nashville, TN 37204
Phone: 615-726-0177
Toll Free: 800-765-0980
Fax: 615-726-3404

Client: _____ **PO#** _____

Address: _____ **Invoice to:** _____

City/State/Zip: _____ **Report to:** _____

Client Invoice Contact: _____ **Project Name:** _____

Client Project Manager: _____ **Reg District (CA):** _____

Telephone Number: _____ **Fax No.:** _____ **Facility ID:** _____

Sampler Name: (Print) _____ **Site Address:** _____

Sampler Signature: _____ **City, State, Zip:** _____

							Preservative								Matrix						Analyze For:																				
Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Ice	HNO_3 (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H_2SO_4 Plastic (Yellow Label)	H_2SO_4 Glass(Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Wastewater	Drinking Water	Sludge	Soil	Other (specify):																					**RUSH TAT (Pre-Schedule)**

Special Instructions:

Method of Shipment: FEDEX

Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by TestAmerica:	Date	Time

Laboratory Comments:

Temperature Upon Receipt:

VOCs Free of Headspace? Y N